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16. ABSTRACT

The research described in th~~is~~e report was concerned with evaluation of alternative approaches to anaerobic digestion, a process commonly used on the residual stream from wastewater treatment. The principal approach studied was that of separating the acid- and methane-forming phases by using two digestion vessels rather than one. The effects of varying temperature and other operating parameters, and of adding enzymes to the process~~,~~ were also investigated.

For continuous-flow, continuously stirred tank reactors, the two-phase process perfomed better than single-stage digestion for all operating conditions. Novel upflow digesters having high solids retention were also evaluated and offered further improvements. The enzyme additions also enhanced two-phase digestion.

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FOREWORD

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water systems. Under a mandate of national environmental laws, the agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. The Clean Water Act, the Safe Drinking Water Act, and the Toxic Substances Control Act are three of the major congressional laws that provide the framework for restoring and maintaining the integrity of our Nation's water, for preserving and enhancing the water we drink, and for protecting the environment from toxic substances. These laws direct EPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Water Engineering Research Laboratory is that component of EPA's Research and Development program concerned with preventing, treating, and managing municipal and industrial wastewater discharges; establishing practices to control and remove contaminants from drinking water and to prevent its deterioration during storage and distribution; and assessing the nature and controllability of releases of toxic substances to the air, water, and land from manufacturing processes and subsequent product uses. This publication is one of the products of that research and provides a communication link between the researcher and the user community.

The research described in this report was concerned with evaluation of alternative approaches to anaerobic digestion, a process commonly used on the residual stream from wastewater treatment. The principal approach studied was that of separating the acid and methane forming phases by using two digestion vessels rather than one. The effects of varying temperature and other operating parameters, and of adding enzymes to the process, were also investigated.

Francis T. Mayo, Director
Water Engineering Research Laboratory

ABSTRACT

Laboratory research was conducted to study the performance characteristics of separate acid- and methane-phase anaerobic sludge digesters and the overall two-phase systems under mesophilic and thermophilic fermentation conditions at several levels of hydraulic flow-through and organic loading rates, culture pH, and feed solids consistency. Chicago municipal wastewater sludges were used as digester feeds. The sludges were chemically and biochemically characterized, and theoretical digestion efficiencies and anaerobic biodegradability factors were determined. Performances of single-stage and two-phase systems using continuous-flow, continuously-stirred tank reactor (CFCSTR) digesters were studied under a variety of comparable operating conditions. The effects of three important variables (pH, hydraulic residence time, and temperature) on acid-phase sludge digestion were determined based on the results of digestion runs conducted according to a factorial experimental design. In a more applied part of the research, novel upflow digesters which were mixed by indigenous gas production and had high solids retention times were used in lieu of the CFCSTR digesters to develop an advanced two-phase system. The study also included investigation of the effects of cellulase-cellobiase pretreatment of the two-phase process sludge feed and of lipase treatment of the acid-phase digester on liquefaction, acidification, and gasification efficiencies.

The CFCSTR two-phase process performed better than CFCSTR single-stage digestion under all operating conditions. The performance of the two-phase process was further enhanced by using the upflow digesters and cellulase-cellobiase pretreatment of the feed sludge in combination with direct lipase treatment of the acid-phase culture.

This report was submitted in fulfillment of Cooperative Agreement No. CR 809982 by the Institute of Gas Technology under the sponsorship of the U.S. Environmental Protection Agency. This report covers the period October 1, 1982, to November 30, 1985, and work was completed as of November 30, 1985.

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ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

SNG -- substitute natural gas
UASB -- upflow anaerobic sludge blanket
CFCSTR -- continuous-flow continuously-stirred tank reactor
Meso -- digester operated at a mesophilic (35°C) temperature
Thermo -- digester operated at a thermophilic (55°C) temperature
Meso-meso -- two-phase system with mesophilic acid- and methane-phase digesters
Meso-thermo -- two-phase system with mesophilic acid-phase and thermophilic methane-phase digesters
Thermo-thermo -- two-phase system with thermophilic acid- and methane-phase digesters
A-P -- acid-phase digester
M-P -- methane-phase digester
HRT -- hydraulic residence time
SCF -- standard cubic feet (dry) at 60°F and 30-in. Hg
SCM -- standard cubic meter (dry) at 15.55°C and 762 mm Hg
VA -- volatile acids
TVA -- total volatile acids as acetic
TS -- total solids
VS -- volatile solids
FS -- fixed solids
TSS -- total suspended solids
VSS -- volatile suspended solids
FSS -- fixed suspended solids
COD -- chemical oxygen demand
CPL -- carbohydrates, protein, and lipids
ΣCPL -- sum of carbohydrates, protein, and lipids
VS_R -- volatile solids reduction
ANOVA -- analysis of variance
ORP -- oxidation reduction potential

SYMBOLS

α -- level of significance
μ -- specific growth rate
K_s -- saturation coefficient

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SECTION 1

INTRODUCTION

Anaerobic digestion is an important aspect of biotechnology because of its applicability to waste treatment, energy conversion, and production of chemicals. In addition, the digestion process can be coupled to appropriate thermal and electrochemical processes to produce electric power, methanol, hydrocarbons, and other useful products.

The conventional digestion system using the single-stage continual-flow, continuously-stirred tank reactor (CFCSTR) digester is perhaps the most commonly used in commercial sludge stabilization. The single-stage CFCSTR process has several disadvantages, however, including the requirement of large, capital-intensive digestion tanks and process instability because of imbalances between the symbiotic fermentation steps of acid formation by acidogenic bacteria (acidogens) and acid-gasification by the more sensitive and slow-growing methane bacteria (methanogens).

A review of the anaerobic digestion literature indicates that an advanced digestion mode in which the acid-production and acid-gasification phases are conducted separately has several advantages over single-stage digestion. Among the principal benefits of this two-phase digestion process are reduced plant capital and operating costs, enhanced stabilization and gasification rates and efficiencies, increased net energy production, and improved process stability and reliability at higher system loadings and shorter hydraulic residence times (HRT's). Although several two-phase digestion studies have been conducted with soluble synthetic substrates and wastes to support some of the claimed benefits of two-phase digestion, there is a paucity of information on process behavior with such particulate feeds as sewage sludge.

First, it is necessary to demonstrate that two-phase digestion of sludge is superior to single-stage digestion under identical operating conditions. The relative advantages (if any) of two-phase digestion over the single-stage process could be a function of such basic operating parameters as HRT, fermentation temperature, and pH; these should be studied. Next, the effects of these important variables on acid- and methane-phase digestion stages need to be delineated. The efficiencies of the conversion of the major organic components of sewage sludge — carbohydrates, proteins, and lipids — by two-phase and conventional digestion at appropriate ranges of HRT and digestion temperature should be compared to establish the superiority of one or the other process. Overall, it is necessary to quantify the benefits of two-phase digestion in terms of gains in conversion rates and efficiencies relative to the baseline performance of single-stage digestion. This project was undertaken to develop this fundamental information by conducting parallel two-phase and single-stage digestion studies, with each process using the same reactor design to obtain a valid comparison. Although several reactor designs are available to conduct anaerobic digestion, the commonly used CFCSTR reactor was selected for the process comparison experiments.

Once the superiority of the two-phase process is demonstrated — an important initial goal — it is also necessary to investigate means of improving this process further to maximize solids stabilization as well as the volumetric gas formation rate at system HRT's that are lower than those of the CFCSTR digesters. Two obvious approaches to accomplishing these goals are 1) to use reactor designs that provide solid residence times (SRT's) that are higher than those of the CFCSTR, and 2) to enhance the reactivity (or biodegradability) of the feed sludge by cost-effective enzyme or chemical treatment. Accordingly, the second part of this research project was concerned with 1) the application of novel upflow digesters that are known to exhibit better performances than those of CFCSTR digesters, and 2) cellulase and lipase treatment of the raw sludge to increase the digestibilities of these two major sludge components.

PROJECT OBJECTIVES

The overall objective of this research was to conduct fundamental and applied studies to demonstrate the application of an advanced digestion process (two-phase digestion) to stabilize and gasify sewage sludges at higher overall rates and efficiencies than are achieved by conventional high-rate digestion.

The research was directed toward developing a better understanding of the fundamental engineering aspects of the acid-forming and methane-forming phases of the overall anaerobic digestion process and investigating selected two-phase process configurations for further development by pilot-plant-scale application later at a selected sewage treatment plant. To address these objectives, the program was divided into two parts: Fundamental studies and applied studies. Specific objectives for each part of the program are described below.

Specific Objectives

Fundamental Studies--

The specific objectives of the Fundamental Studies were to conduct bench-scale anaerobic digestion experiments and —

- To compare two-phase and conventional single-stage, high-rate anaerobic digestion processes at mesophilic and thermophilic culture temperatures at selected HRT's

- To develop basic information on the effects of key operating variables on acid-phase digester performance with a selected sewage sludge.

Applied Studies--

The specific objectives of the Applied Studies were —

- To conduct a bench-scale investigation of an advanced two-phase digestion mode using novel upflow acid- and methane-phase digesters and interphase culture recycling to identify the best two-phase digestion process configuration for pilot-scale testing later in a sewage treatment plant

- To study the effect of sludge pretreatment by selected cellulase and lipase enzyme systems on two-phase process performance at a short hydraulic residence time.

SECTION 2

RESULTS AND CONCLUSIONS

Several important aspects of single-stage and two-phase anaerobic digestion were investigated during this research. The results of the experimental work showed that under comparable operating conditions, the two-phase sludge digestion process was more stable and exhibited higher conversion efficiencies and rates than single-stage digestion. Theoretical and kinetic analyses of single-stage and two-phase processes supported these experimental observations. The performance of the two-phase digestion process could be improved considerably by the use of high-SRT upflow reactors in lieu of CFCSTR digesters, or by pretreatment of the digester feed by a commercial cellulase-cellobiase system and direct lipase treatment of the acid-phase digester. It was observed that, although sludge liquefaction and acidification were enhanced at a higher digester temperature, the thermophilic acetogens and methanogens were inhibited by certain unidentified products of protein and lipid degradation.

Only 59% of the sludge volatile solids were anaerobically biodegradable, and complete conversion of the biodegradable material was achieved by two-phase digestion.

The following are specific results and conclusions from work on the specific research topics listed below.

KINETIC ANALYSES OF SINGLE-STAGE AND TWO-PHASE DIGESTION

1. Kinetic analysis based on the available information indicated that, in theory, the rate of production of volatile acids in single-stage CFCSTR anaerobic digesters is higher than the rate of conversion of volatile acids unless the digester is operated at a fairly high HRT — usually 20 days or higher. Single-stage digestion at lower HRT's leads to an imbalance between volatile acids production and volatile acids conversion, resulting in acids accumulation and inhibition of acidogenic and/or methanogenic fermentation(s). Also digester operation at unduly high HRT's is tantamount to maintenance of the acidogenic bacteria in the stationary or the endogenous growth phases, which leads to the deceleration of the substrate hydrolysis and acidification processes. Two-phase fermentation, which provides for separate culturing of the kinetically dissimilar digester organisms, has the advantages of maximizing the rates of volatile solids degradation and volatile acids formation and minimizing volatile acids conversion (methanogenesis) in the acid-phase digester. Methanogenesis, on the other hand, is maximized and the acid formation rate is minimized in the methane digester. Thus, in two-phase fermentation, both groups of digester organisms could be maintained in

the exponential growth phase and the probability of unbalanced acidogenic-methanogenic fermentation and its inhibition is minimized. The two-phase process thus should afford higher sludge stabilization rates and efficiencies at lower HRT's and higher loading rates than are feasible with single-stage digestion.

CHEMICAL CHARACTERISTICS OF DIGESTER FEEDS

1. About 80-95 wt % of total solids (TS) content of the Chicago sludge used for the digestion studies was insoluble particulate matter with the balance being soluble material. On the average, about 8 wt % of the TS was soluble inorganics, 21 wt % (of TS) was insoluble inorganics (fixed solids), 7 wt % (of TS) was soluble organics, and 64 wt % (of TS) was insoluble organics. The total COD of the sludge solids averaged 1.5 g COD/g VS. About 93% of the sludge COD was due to particulate organic matter. Of the total nitrogenous matter, 85-91% was particulate proteinaceous material. The average protein, carbohydrate, and lipid contents of the feed sludge lots were about 33 wt %, 24%, and 27 wt % of the VS, respectively. On the average, about 83 wt % of organics could be accounted for by proteins, lipids, and carbohydrates. The chemical characteristics of the feed indicated that the success of the digestion process would be dictated to a large extent by the ability of the digester culture to hydrolyze proteins and lipids and metabolize the hydrolysis products to produce acetate or other methanogenic substrates.

2. Decomposition of the feed sludge during storage and delivery to the digester could be minimized by refrigerated storage. It was observed that certain indicator chemical characteristics of the digester feed slurry remained virtually unchanged and stable during refrigerated storage at 2° to 4°C, as indicated by a time-series analysis of the feed reservoir contents for TS, VS, and individual volatile acids during a worst-case storage period of 8 days.

THEORETICAL EFFICIENCIES AND CHEMICAL AND BIOCHEMICAL REACTIVITIES OF DIGESTER FEEDS

1. The theoretical total gas (biogas) and methane yields of the Hanover Park wastewater sludge, as estimated from the elemental analysis, theoretical carbonaceous COD, and calorific value, were within a few percentage points of each other. The total biogas and methane yields of this feed sludge were 0.50 and 0.79 SCM/kg VS reacted (8.0 and 12.6 SCF/lb VS reacted), respectively. In contrast to these observations, the analytical COD's (in terms of kg of measured total and carbonaceous COD's per kg of sludge VS) of the different sludge lots collected at various times of the year were significantly different from each other; consequently, theoretical methane yields of different lots based on measured carbonaceous COD's differed from each other by as much as about 38%. The analytical carbonaceous COD content and the analytical COD-based methane yields of the Hanover Park sludge were related to the sum total of the masses of total carbohydrate, crude protein, and lipids in the sludge (ΣCPL). It was found that the theoretical methane yield varied directly as the ΣCPL of the sludge.

2. Comparison of the elemental-composition-based and analytical (directly measured) COD's of the raw sludge showed that about 59% of the sludge volatile solids (VS) was chemically oxidizable under the COD test conditions.

3. Results of long-term batch anaerobic digestibility potential (ADP) tests indicated maximum expected biogas and methane yields of 0.46 and 0.32 SCM/kg VS added (7.3 and 5.1 SCF/lb VS added) from mesophilic digestion of the Hanover Park wastewater sludge. These gas yields provided a VS reduction of 48%. Results of the ADP test showed that about 80 wt % of the sludge VS was rapidly biodegradable and about 20 wt % was relatively recalcitrant to mesophilic (35°C) anaerobic digestion.

4. A comparison of the theoretical and the ADP methane yields indicated that about 58% of the sludge VS was anaerobically biodegradable under mesophilic (35°C) conditions. This biodegradability factor compared well with the observation that about 59% of the sludge carbonaceous VS was chemically oxidizable. Thus, it appeared that the chemical oxidizability of the carbonaceous VS could be a good measure of anaerobic biodegradability.

5. There was a high degree of correlation between influent and effluent VS and total carbon concentrations; VS reductions calculated on the bases of mass-flow of gaseous carbon out and mass-flow of feed carbon in seemed to be more accurate than those calculated by other methods.

SINGLE-STAGE CFCSTR DIGESTION

1. A comparison of the performances of mesophilic single-stage CFCSTR runs conducted at 15, 7, and 3-day HRT's indicated that optimum process performance would be expected at an HRT of 7 days.

2. With single-stage CFCSTR digesters, the best thermophilic performance was obtained at an HRT of 15 days. Gas and methane yields and organic reductions decreased and volatile acids accumulation increased as the digester HRT was decreased from 15 to 7 to 3 days. Inhibitory levels of volatile acids were observed at the 7 and 3-day HRT's.

3. There was clear evidence of the occurrence of unbalanced acidogenic-methanogenic fermentations in the single-stage CFCSTR processes at the 3-day HRT under the mesophilic and thermophilic conditions.

4. Comparison of steady-state performances of single-stage CFCSTR mesophilic and thermophilic runs under the comparable HRT and loading rate conditions showed that higher methane yields and production rates were obtained at the thermophilic temperature at all operating conditions tested (HRT's of about 15, 7, and 3 days and loading rates of about 2, 7, and 15 kg VS/m^3-day).

5. The highest thermophilic methane yield and methane production rate of 0.28 SCM/kg VS added (4.5 SCF/lb VS added) and 1.8 vol/vol-day, respectively, observed in the single-stage CFCSTR systems were 24% and

12% higher than the highest observed single-stage CFCSTR mesophilic methane yield and methane production rate.

6. Volatile acids concentrations of the single-stage CFCSTR thermophilic digester effluents were in all cases higher than those of the single-stage CFCSTR mesophilic digester effluents, suggesting that higher degrees of hydrolysis and acidification occurred during thermophilic operation.

7. Even though the volatile acids concentrations were high in the thermophilic CFCSTR, the pH levels remained high due to higher alkalinities and ammonia nitrogen concentrations in the thermophilic CFCSTR than in the mesophilic CFCSTR.

8. Although the single-stage thermophilic digesters effected enhanced hydrolysis and acid production, gasification of the volatile acids was not similarly enhanced, with the result that high concentrations of unconverted acids accumulated and emanated with the thermophilic effluents.

9. During single-stage CFCSTR digestion, carbohydrate conversion was highest (44%) during mesophilic operation at the lowest HRT (3 days); in contrast, the highest carbohydrate conversion (25%) during thermophilic operation was observed at the highest HRT (15 days). Thermophilic carbohydrate reductions in single-stage CFCSTR's were lower than those at the mesophilic temperature at all test HRT's.

10. Crude protein reductions in thermophilic single-stage CFCSTR digesters at 15 and 7-day HRT's were about double those in the mesophilic single-stage CFCSTR digesters; however, thermophilic and mesophilic protein conversions were comparable at the lowest HRT.

11. During single-stage CFCSTR digestion, lipid reductions at the thermophilic temperature were higher than those at the mesophilic temperature at all HRT's.

CFCSTR TWO-PHASE DIGESTION

Acid-Phase Digestion

1. The mesophilic and thermophilic acid-phase digesters exhibited enhanced volatile acids production and gas-formation rates as the HRT was decreased from 2 to 0.9 days and the loading rate was increased from 2 to 15 kg VS/m^3-day.

2. The "natural" pH of the mesophilic and thermophilic acid-phase digesters stabilized at about 6.6.

3. There was no evidence of hydrogen accumulation in the head gases except during mesophilic operation at an HRT of 0.9 days. These observations (coupled with the fact that all acid-phase runs exhibited high methane-content gases) indicated that the rate of hydrogen utilization by the

syntrophic — and carbon dioxide-reducing — methane bacteria exceeded the rate of hydrogen production during oxidation of the sludge hydrolysates.

4. It was apparent that the wash-out (or critical) HRT of the syntrophic methane formers was less than 1 day.

5. Gases from the mesophilic and thermophilic acid digesters contained more nitrogen than those of the methane digester, suggesting that substrate oxidation during acidogenesis was coupled to nitrogen oxides reduction (denitrification).

6. The ΣCPL [sum total of the masses of total carbohydrate (C), crude protein (P), and lipids (L)] reductions in the mesophilic acid digester were higher than those in the mesophilic methane digester at all HRT's and loading-rates, indicating that liquefaction of organics was predominant in the acid digester. Conversely, ΣCPL reductions in the mesophilic acid digesters were lower than those in the thermophilic methane digester, suggesting continued and enhanced liquefaction of the organics under the thermophilic conditions of the latter digester.

7. Carbohydrate and lipid reductions in the mesophilic acid-phase digester decreased as the HRT was decreased from 2 to 0.9 days.

8. Acetate was the predominant volatile acid in the mesophilic and thermophilic acid digesters, followed by propionic and isovaleric/butyric.

Methane-Phase Digestion

1. Total gas and methane yields and the gas-phase methane content decreased, and their production rates and acids accumulation increased as the HRT of the mesophilic methane digester was decreased from 13 to 5 to 2 days.

2. Surprisingly, both gas and methane yields and production rates, and volatile acids accumulations, increased as the HRT of the thermophilic methane digester was reduced from 13 to 5.5 days; however, all these performance parameters exhibited lower values as the HRT was decreased from 5.5 to 2 days. Thus, a methane digester HRT of 5.5 days seemed to be optimum for thermophilic methanogenesis.

3. About 86% of the meso-meso two-phase system methane production was derived from the mesophilic methane digester when the ratio of the methane digester HRT to the system HRT was 0.87; this percentage dropped as the HRT ratio was decreased. Thermophilic methane digesters fed with mesophilic acid digester effluents showed the same trend as above, but exhibited a lower proportion of system methane production when operated in series with a thermophilic acid digester.

4. The methane contents and bicarbonate alkalinities of both the mesophilic and thermophilic methane digesters were considerably higher than those of the acid digesters. The bicarbonate alkalinity increased significantly during methane fermentation; enhancement of the bicarbonate alkalinity

had the effect of scrubbing the gas-phase CO_2, thereby enhancing the gas-phase methane content of the methane digester.

5. For a given HRT and loading-rate, the thermophilic methane digester exhibited much higher acids accumulation and ammonia-nitrogen concentration than the mesophilic methane digester. Accumulation of propionate was higher than that of any other volatile acids.

Overall System Performance

1. The results of meso-meso, meso-thermo, and thermo-thermo two-phase digestion studies showed that, for 15-day and 3-day system HRT's, the meso-meso operations were better than the meso-thermo or thermo-thermo operations in terms of gas and methane productions and VS and organic reductions. For a 7-day HRT, the meso-thermo two-phase system was better than the meso-meso or the thermo-thermo operation in terms of methane yield and VS reduction.

2. The meso-thermo and thermo-thermo two-phase processes had much higher effluent volatile acids concentrations than the meso-meso two-phase process.

3. Although liquefaction and acidification were enhanced under thermophilic conditions, the gasification reactions were not similarly enhanced.

PROCESS COMPARISON: CFCSTR SINGLE-STAGE VERSUS CFCSTR TWO-PHASE

Process Comparison at a 15-Day System HRT

1. Both the meso-thermo and the meso-meso two-phase processes were superior to the mesophilic or thermophilic single-stage digestion in all respects.

2. The meso-meso two-phase run exhibited the best performance; it afforded the highest methane yield of 0.41 SCM/kg VS added, a VS reduction of 54%, and a ΣCPL conversion efficiency of 57%, indicating complete conversion of biodegradable organics. By comparison, the best single-stage methane yield and VS reduction were 0.28 SCM/kg VS added and 39%, respectively. The above meso-meso two-phase methane yield was 82% of the theoretical methane yield, which was based on 100% recovery of the feed carbon in the digester gases.

Process Comparison at a 7-Day System HRT

1. The meso-thermo two-phase system exhibited better performance than the meso-meso and the thermo-thermo two-phase and the single-stage mesophilic and thermophilic digestion processes in terms of gas and methane yields, methane content of gas, and VS (total and biodegradable) and ΣCPL reductions; however, the volatile acids concentration in the effluent of the meso-thermo system was higher than those of the other systems. The next best performance was exhibited by the meso-meso two-phase process; the effluent acids concentration of this process was one-tenth of the concentration of the meso-thermo effluent.

2. The performance of the thermo-thermo two-phase process was worse than those of the meso-thermo and meso-meso two-phase processes.

3. The methane-phase gas production declined sharply when it was charged with thermophilic acid-digester effluent, indicating that thermophilic metabolites could have retarded the acetogenic and/or methanogenic bacteria.

4. Two-phase digestion effected higher lipid and carbohydrate degradations than single-stage digestion. Protein and lipid conversions were enhanced under thermophilic conditions. Carbohydrate reduction was enhanced under mesophilic conditions. The lipid degradation efficiency was higher than those of proteins and carbohydrates.

Process Comparison at a 3-Day System HRT

1. The thermo-thermo and the meso-meso two-phase systems exhibited higher methane yields and production rates and VS and organic reductions than the mesophilic and thermophilic single-stage digesters. However, at the 3-day HRT, the meso-meso two-phase system was better than the thermo-thermo process.

2. Volatile acids accumulations (2000-3200 mg/L) in the single-stage digesters were double those of the two-phase systems showing that, whereas unbalanced digestion occurred in the former process at a 3-day HRT, a more balanced acidogenic-methanogenic fermentation was experienced in the latter fermentation mode.

3. At a 3-day system HRT, the methane yield of the meso-meso two-phase process was 102% higher than that of single-stage mesophilic digestion; this positive methane yield differential at the 3-day HRT was higher than those observed at the 7 and the 15-day HRT's. Thus, the benefits of two-phase fermentation were more evident at the lowest HRT.

CHARACTERISTICS OF THERMOPHILIC DIGESTION

1. Volatile acids gasification efficiency at the thermophilic temperature was lower than that under mesophilic digestion conditions.

2. Under thermophilic conditions, the efficiencies of volatile acids conversion by two-phase digestion was higher than those by single-stage digestion.

3. Relative to mesophilic (35°C) digestion conditions, the thermophilic (55°C) digestion temperature enhanced the hydrolysis of sludge particles and acidification of the hydrolysate but retarded acetogenic conversions of propionate, branch-chain fatty acids, and caproate and methanogenic conversions of acetate and hydrogen and CO_2.

4. Thermophilic degradation products of proteins and lipids seemed to be inhibitory to thermophilic acetogens and methanogens.

5. For comparable operating conditions, buffer capacities of the methane-phase digesters of the two-phase systems were higher than those of the single-stage digesters, and this positive alkalinity differential increased as the system HRT decreased from 15 to 7 to 3 days. The two-phase process was thus more stable than the single-stage digester, and the relative stability of the former process increased as the system HRT decreased.

EFFECTS OF pH, HRT, AND TEMPERATURE ON ACID-PHASE DIGESTION

Mesophilic Acid-Phase Runs: pH and HRT Effects

1. The optimum pH for mesophilic acidogenesis of the Hanover Park sludge was between 5.5 and 6.2; gas and acid productions were maximized within this range. At an HRT of 2 days, protein, lipid and ΣCPL reductions were higher at pH 5 than at pH 7; carbohydrate reductions at these two pH's were about the same. At an HRT of 1.3, pH 7 effected a higher degree of acidogenesis than pH 5. The worst acid-phase performance was obtained at an HRT of 1.3 days and a pH of 5, which was a combination of a low HRT and a low pH. There was hydrogen in the digester gas at an HRT of 1.3 days and a pH of 5.

2. At pH 7, the efficiency of acidogenesis was higher at an HRT of 2 days than at 1.3 days. At pH 5, methane yield and production rate were higher at an HRT of 1.3 days than at an HRT of 2 days, whereas acids productions at these two HRT's were about the same.

Thermophilic Acid-Phase Runs: pH and HRT Effects

1. Optimum thermophilic acid-digester performance was obtained at a pH of 6.

2. Carbohydrate, protein, lipids and ΣCPL reductions, and gas and volatile acids productions, were higher at pH 7 than at pH 5.

3. At pH's 7 and 5, gas and volatile acids productions were higher at an HRT of 2 days than at an HRT of 1.3 days.

4. As with mesophilic acid-phase digestion, the worst thermophilic acid-phase digester performance was observed at the lowest HRT of 1.3 days and at the lowest pH of 5.

Temperature Effects

1. Gas and methane yields and production rates from the thermophilic acid digesters were lower than those from the mesophilic acid digester under all test operating conditions; however, the reverse was true for volatile acids production. These observations indicated that the activities of the syntrophic methane formers were probably inhibited under thermophilic conditions.

Statistical Inference from Analysis of Variance (ANOVA) of the Factorial Experiment

1. The culture pH had a strong influence on the conversion of VS and the major organic components of carbohydrate, protein, and lipids; these conversions increased as the pH was increased to the optimum value.

2. Increases in culture temperatures and HRT tended to increase carbohydrate conversion; however, the effect of each variable could not be separated because of large temperature-HRT interaction.

3. Increase in acid-digester HRT tended to increase protein and lipid reductions.

4. The digester HRT, temperature, and culture pH each independently influenced (and increased) acid-digester gas yield; the gas yield decreased as the HRT and culture temperature increased, and it increased as the culture pH was increased.

5. Overall, enhanced hydrolysis and acidification were achieved at the thermophilic temperature and at a pH of about 6.

OPTIMUM OPERATING CONDITIONS FOR TWO-PHASE DIGESTION

1. Based on the process comparison and parametric-effect acid-phase digestion studies, the following operating conditions were regarded optimum for two-phase anaerobic digestion of Hanover Park sludge:

   - A culture temperature of 35°C
   - A system HRT of 7 days (a 2-day HRT for acid digester and a 5-day HRT for methane digester)
   - A feed VS concentration of 50 g/L
   - A pH of about 6.6 for the acid digester, obtainable without pH control
   - A pH of 7 or higher for the methane digester.

ADVANCED MESOPHILIC TWO-PHASE DIGESTION WITH NOVEL UPFLOW REACTORS

1. Methane yield from the mesophilic upflow two-phase system was 17% higher than that from the CFCSTR two-phase system with both systems operated at an HRT of 7 days and a system loading rate of about 7 kg/m^3-day; this increase in methane yield reflected the reactor effect.

2. Volatile acids production in the upflow acid-phase digester was about three times that of the CFCSTR acid digester. There was no evidence of acetogenesis occurring in the acid digester.

3. The upflow two-phase digestion system exhibited higher VS, protein, carbohydrate, and lipid reduction than the CFCSTR two-phase digestion process.

4. Acetate and the higher volatile acids were readily converted within the bottom one-half of the culture depth of the upflow methane digester. Acetogenesis, acetate conversion, and syntrophic methane fermentation were the predominant reactions in the methane digester.

5. Recycling of the upflow methane digester effluents to the upflow acid digester enhanced methane and nitrogen gas yields and production rates and the volatile acids formation rate, indicating that this recycling had the effect of enhancing sludge hydrolysis and acidification.

ADVANCED THERMOPHILIC TWO-PHASE DIGESTION WITH NOVEL UPFLOW REACTORS

1. During meso-thermo upflow two-phase digestion of Hanover Park sludge, the thermophilic methane-phase digester produced more acids than the mesophilic acid digester, and system gas yield was low under these conditions. Two-phase system performance did not improve when the upflow acid digester temperature was changed from 35°C to 55°C.

2. There was little evidence of acetogenic and methanogenic activities in the upflow methane digester.

3. The inhibitory effects of thermophilic metabolities on aceto-methanogenesis were more severe on the high-SRT upflow methane digester than on the CFCSTR methane digesters. The reason for this enhanced inhibition could be that the high-SRT upflow digester contained a larger reservoir of inhibitor-producing substances than the CFCSTR digester, which experiences continual flushing of the inhibitors.

THERMO-THERMO-THERMO THREE-STAGE DIGESTION

1. A three-stage system consisting of an upflow acid digester, an upflow methane digester, and a CFCSTR methane digester performed better than the meso-thermo or the thermo-thermo upflow two-phase systems primarily because the CFCSTR methane digester exhibited considerably higher gasification efficiency than the upflow methane digester.

2. It was observed that a thermophilic temperature of 60°C was detrimental to methane fermentation of thermophilic acid-digester effluents; gas production practically ceased at 60°C.

3. Considerably improved performance of a thermo-thermo upflow two-phase system could be obtained when a prolonged enrichment and acclimation period was used, and when a mixed Downers Grove primary/Stickney activated sludge was substituted for the Hanover Park sludge. Apparently, the degree of thermophilic inhibition could depend on the nature and source of the raw sludge.

MESO-MESO TWO-PHASE CFCSTR DIGESTION OF ENZYME-TREATED SLUDGE

1. Cellulase-cellobiase treatment of the feed sludge and lipase treatment of the acid-phase digester increased the methane yield from the mesophilic CFCSTR two-phase system by about 23% over that obtained from digestion of the untreated raw sludge; this increase was significant at the system HRT of 3 days used to evaluate the effect of enzyme treatment.

2. Carbohydrate reductions by the acid-phase digester and the overall two-phase system were 50% and 64%, respectively, with cellulase-cellobiase treatment compared with the corresponding reductions of 16% and 50% obtained without enzyme treatment, indicating that the commercial enzyme system was effective in hydrolyzing sludge carbohydrates.

3. Lipid reductions by the methane-phase digester and the overall two-phase process were 36% and 39%, respectively, with lipase treatment compared with the corresponding reductions of 9% and 27% obtained without such treatment, indicating that the commercial lipase was effective in hydrolyzing the sludge lipids.

4. Two-phase system effluents contained lower volatile acids concentrations when the system feed sludge was enzyme-treated than those obtained without any enzyme treatment. This performance, considered together with the observed enhancement of methane content, yield, and production rate achieved by enzyme treatment, seemed to indicate that enzyme treatment of sludge had beneficial effects on the acidogenic, acetogenic, and methanogenic organisms.

SECTION 3

RECOMMENDATIONS

The process-comparison studies of this project demonstrated that the two-phase digestion process can exhibit performances that are superior to those of the single-stage digestion process under a comparable set of operating conditions. These studies should be repeated with larger scale pilot systems, but under one set of selected operating conditions and with CFCSTR digesters and concentrated sludge feeds.

In this research the best system performance was exhibited by a two-phase system that utilized high-SRT novel upflow digesters. It is recommended that engineering studies be undertaken to investigate the flow regimes, solids-retention characteristics, etc., of digesters of unconventional design. Considerable work remains to be done to develop the structural details of efficient digestion reactors.

This research indicated that the biochemical steps of acetogenesis and aceticlastic and syntrophic methane fermentation could be severely retarded during thermophilic fermentation of sewage sludge. It is recommended that a separate investigation of the causative factors underlying this problem be initiated.

The effect of temperature on acid and methane fermentation should be studied in more detail than was accomplished in this project.

Stabilization (reduction) of volatile solids is an important criterion for evaluating digester performance in a municipal wastewater treatment plant. Volatile solids reduction calculation, which is needed for this evaluation, is commonly based on an outmoded procedure. Unrealistic and inaccurate results are obtained when determinations of residual sludge mass for ultimate disposal are based on such calculation methods as the one outlined in the Water Pollution Control Federation (WPCR) Manual of Practice (MOP) No. 16. There is a need to develop a better, more accurate method for the determination of volatile solids reduction during anaerobic digestion.

SECTION 4

BACKGROUND

UTILITY OF ANAEROBIC DIGESTION

Waste Treatment Application

One of the widely used unit processes in sewage and industrial wastewater treatment is anaerobic digestion. During its more than 100 years of commercial application, anaerobic digestion has proved to be a cost-effective disposal process because of the following characteristics:

- The capability to stabilize large volumes of dilute organic slurries (sludge) at relatively low cost
- The capability to generate medium-Btu fuel gas from low-grade feed carbon
- Low microbial cell mass production
- The capability to produce stable and odor-free solid residues of good fertilizing value
- A high kill rate of pathogenic organisms and viruses.

Anaerobic digestion is preferred to alternative biological and thermochemical sludge stabilization methods that are energy-intensive and have unfavorable environmental impacts. Increased application of this process to waste stabilization is expected, both to conserve energy and to help meet new and stringent pollution abatement standards.

Sludge treatment and disposal are among the most difficult problems in wastewater treatment and represent as much as 25% to 30% of the capital and operating costs of a sewage treatment plant.[1,2] Also, published data indicate that about 40% of the total cost of sludge handling and disposal is incurred in operating the digestion system.[3,4] Any improvement of the conventional sludge digestion process could thus result in substantial savings in waste disposal costs.

Energy-Production Application: Methane From Wastes

One attractive feature of anaerobic digestion is its ability to convert organic (and inorganic) carbon in the feeds to a product gas stream high in methane, which has a high demand as a clean fuel. This aspect of the process has prompted several investigators to advocate its application for the simultaneous stabilization and gasification of municipal, industrial, and agricultural wastes.[5] There was an upsurge of interest in applying anaerobic digestion for methane (substitute natural gas or SNG) production from biomass and waste since the advent of the "energy crisis" in the early 1970's. The impact of SNG from waste alone could be significant. For example, it is estimated that in a city of 1 million people, 0.3 to 0.6 million SCM/day of SNG may be obtained by digesting municipal sludge and refuse alone. This quantity of SNG could satisfy 5% to 15% of the community's natural gas demand.

Nationwide, about 180 million metric tons of municipal refuse, 80 million metric tons of industrial waste, 3 billion metric tons of agricultural wastes, and 6 million metric tons of sludge solids are currently produced annually. These wastes represent a renewable source of nonfossil carbon which, instead of causing enormous land, air, and water pollution problems, could be converted by anaerobic digestion to produce substantial amounts of supplemental SNG.

Energy-Production Application: Methane From Biomass

Because of the ability of anaerobic digestion to generate fuel gases (methane and hydrogen), an entirely new line of digestion process application is developing for small- and large-scale conversion of biomass and other high-moisture organic feedstocks to SNG.[5-8] Several authors have pointed out the potential of biomass as a plentiful source of renewable energy for both the developing and developed countries. It is recognized that biomass and waste could supply up to 15% of U.S. energy needs by the end of this century via gasification or other conversion schemes,[5] and that anaerobic digestion will play a major role in producing gaseous fuels from biomass.[6,7] The feasibility of SNG production by anaerobic digestion has already been demonstrated for such biomass species as grass, algae, marine giant kelp, and water hyacinth.[9-16] The energy-production application of anaerobic digestion may in time exceed its classical waste treatment application in terms of plant capacity and investment.

Chemical Production

Considerable work has been done to demonstrate that certain novel configurations of the anaerobic digestion process can be employed to produce organic acids from biomass and wastes in reasonable yields with the residue from the acid-production process converted to generate methane.[17,18] Since the prices of the organic acids are about one order of magnitude higher than that of methane on a mass basis, the economics of the digestion process improve significantly with such dual applications.

CONVENTIONAL SLUDGE DIGESTION PROCESSES

Originating from the rather crude septic-tank system and the Imhoff tank, the anaerobic digestion process has evolved through a series of modifications into the high-rate digestion process now employed by many waste-treatment plants. The most widely used process designs are the so-called "standard-rate" (or low-rate) and "high-rate" digestion systems.

Standard-Rate Digestion

Standard-rate digesters are unmixed, covered tanks with hydraulic residence times (HRT's) of 30 to 60 days. These digesters are fed intermittently at organic loading rates of 0.5 to 1.6 kg VS/m^3-day.[19] A state of stratification exists in these tanks, and contact between influent organics and the digesting organisms is limited. Consequently, standard-rate digestion is frequently a diffusion-controlled process.

High-Rate Digestion

High-rate digestion differs from the standard-rate process in that continuous or more frequent feeding and mixing of the raw and digesting sludge are used. Mass transfer, therefore, is theoretically not a limiting factor in high-rate digestion kinetics. This allows process operation at reduced HRT's of 15 days or less; loading rates of 1.6 to 3.2 kg VS/m^3-day for high-rate digester designs are common.[19]

Anaerobic Contact Process

An advanced high-rate digestion system is the anaerobic contact process which is comprised of a high-rate digester, a degasser (optional) and an anaerobic settler. Effluent from the mixed high-rate digester is settled in the settler and a fraction of the settler underflow is recycled to the high-rate digester to maintain a higher biological population. As a result of cell mass recycling, lower hydraulic residence times (9 to 12 days) are possible in the contact process. However, the anaerobic contact process is more suitable for dilute wastes; it is seldom used to stabilize municipal sewage sludge.

Two-Stage Digestion

Standard-rate digesters are single-stage systems that provide for digestion, supernatant separation and sludge concentration, and even digested sludge storage, all in the same tank.[19,20] Aside from the slower kinetics, standard-rate digesters suffer from the disadvantage that valuable heated digester space has to be provided for supernatant separation and storage. Also, the supernatant withdrawn from standard-rate digesters is high in biochemical oxygen demand (BOD) and suspended solids, probably because hindered settling conditions prevail in these tanks.[21] These difficulties are overcome in the "stage-digestion" system in which the fermentation process is accomplished separately from supernatant separation, sludge concentration, and storage. In a common configuration of the stage-digester system, the first digestion tank is mixed and is maintained at the optimum digestion temperature. The second tank receives digested sludge from the first tank and holds it in a quiescent condition to permit a better solid-liquid separation and a polishing treatment of the supernatant liquor.[21,22] The "secondary stage" is often a covered, unheated tank where the digestion process initiated in the first tank continues to "technical completion" at a much slower rate.[21] In some systems an uncovered third tank may also be provided for winter and/or operational storage.

DISADVANTAGES AND LIMITATIONS OF CONVENTIONAL DIGESTION

It is well known that the overall anaerobic digestion process is mediated by at least three different dominant groups of anaerobes — acidogenic, acetogenic, and methanogenic organisms — that are responsible for liquefaction, acetate formation, and methane production. These microbial groups are different from each other in terms of physiology, metabolic characteristics, growth kinetics, environmental requirements, and sensitivity to physical and chemical environmental stresses and toxicity. The conventional digestion process provides for the culturing of these diverse groups of organisms under the same environmental conditions, and no attempt is made to optimize the separate microbial phases. The single-stage conventional digesters harboring mixed microbial phases are designed to accommodate the rate-limiting biochemical step which is frequently that of the methanogenic population. This design philosophy has led to the use of high HRT's, low flowthrough rates, large digestion tanks, and high capital and operating and maintenance costs. Digester operation at low flow rates results in low gas and methane production rates and causes the relatively fast-growing acidogenic organisms to operate at lower growth rates associated with the declining or the stationary growth phases. In addition, considerable difficulties are encountered in mixing large digestion tanks. Lack of adequate or "complete" mixing gives rise to scum formation, sludge deposits, and accumulated incrustations, all of which have the ultimate effect of reducing the active or effective volume of the digester. It has been reported that up to 60% of the digester volume could be "dead" space.

A serious limitation of the conventional single-stage digestion is the occurrence of unbalanced acidogenic and methanogenic fermentations in response to process operation at high organic loading rates. This is illustrated by the data in Table 1.[23] The data presented in this table show that as the loading rate on the mesophilic sludge digester was increased, methane yield and methane content of the digester gases decreased while the digester volatile acids concentration increased. Stable and balanced acidogenic and methanogenic fermentations occurred up to a loading rate of about 1.9 kg VS/m^3-day as indicated by the high methane yields, low residual VA's and high VS stabilization efficiencies. At higher loadings, liquefaction and acidification predominated over methane formation as indicated by the high VA concentrations, lower methane yields and concentrations, and low VS reduction efficiencies. Occurrence of high acids concentration had the effect of inhibiting the methanogenic bacteria.

The above observations indicate that single-stage digesters have an upper limit for the loading rate which is maintained in commercial digesters by increasing the HRT and utilizing dilute feed slurries or both. Low HRT's and high loading rates which decrease digester size and capital and operating costs and increase net energy production (due to lower heating requirement) cannot be applied to the single-stage CFCSTR digester because these operating conditions lead to unbalanced acidogenic and methanogenic fermentations resulting in dominant acidogenic activity, volatile acids accumulation, and inhibition of methane fermentation.

TABLE 1. STEADY-STATE PERFORMANCE OF CONVENTIONAL SINGLE-STAGE MESOPHILIC (35°C) DIGESTION OF SEWAGE SLUDGE IN CFCSTR REACTORS

Loading rate, kg VS/m^3-day	Methane content of digester mol %	Methane yield, SCM/kg VS added	Digester volatile acids, mg/L	VS stabilization efficiency, %
1.28 (0.08)*	72.7	0.300	100	41.3
1.44 (0.09)	69.5	0.310	80	45.5
1.92 (0.12)	70.1	0.290	100	42.7
5.45 (0.34)	57.7	0.076	1370	15.5
9.13 (0.57)	51.1	0.047	3220	11.6

* Numbers in parentheses are the loading rates in lb VS/ft^3-day.

PROCESS IMPROVEMENT NEEDS AND APPROACHES

In view of the important role of anaerobic digestion in sewage sludge stabilization and energy conversion from various types of organic materials, it is necessary to develop an innovative and alternative anaerobic digestion technology that is capable of efficient and rapid-rate conversion of substrates and of exhibiting higher net energy production efficiency.* The conventional digestion processes are not capable of meeting these needs.

Innovative digestion systems necessarily incorporate advanced reactor designs and fermentation modes that permit process operation with concentrated feeds at lower HRT's and higher loading rates than are possible with the conventional high-rate process configurations. Achievement of these objectives, of course, means reduction in capital and operating costs and enhanced net energy (surplus methane) production.

Several approaches could be used to improve the anaerobic digestion process. One approach is to develop and apply novel digestion reactors that retain substrate and microbial solids for residence times significantly higher than the HRT. For biochemical processes that are mediated by several microbial phases, a staged fermentation system, referred to as "two-phase" digestion, that permits separate optimization of the dominant microbial reactions, is expected to exhibit superior performance. Thus, a unified approach to process improvement would involve separate optimization of the liquefaction-acidification and methanation phases with an appropriate novel reactor design selected for each fermentation phase.[24,25]

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* Net energy production efficiency = (Energy value of digester methane — External thermal and electrical energies)/(Energy value of digester methane).

Novel Process Concepts

Various approaches can be envisioned to enhance the efficiency, kinetics, and stability of the anaerobic digestion process. Chemical or enzymatic hydrolysis of the particulate feed and utilization of genetically improved microorganisms, for example, could improve the process, but these methods do not seem to be cost-effective or practical at this time. Engineering approaches including application of advanced operating or fermentation modes and utilization of novel reactors are feasible and merit development for digestion process improvement in the near term. Thus, a two-part approach, as outlined below, is needed:

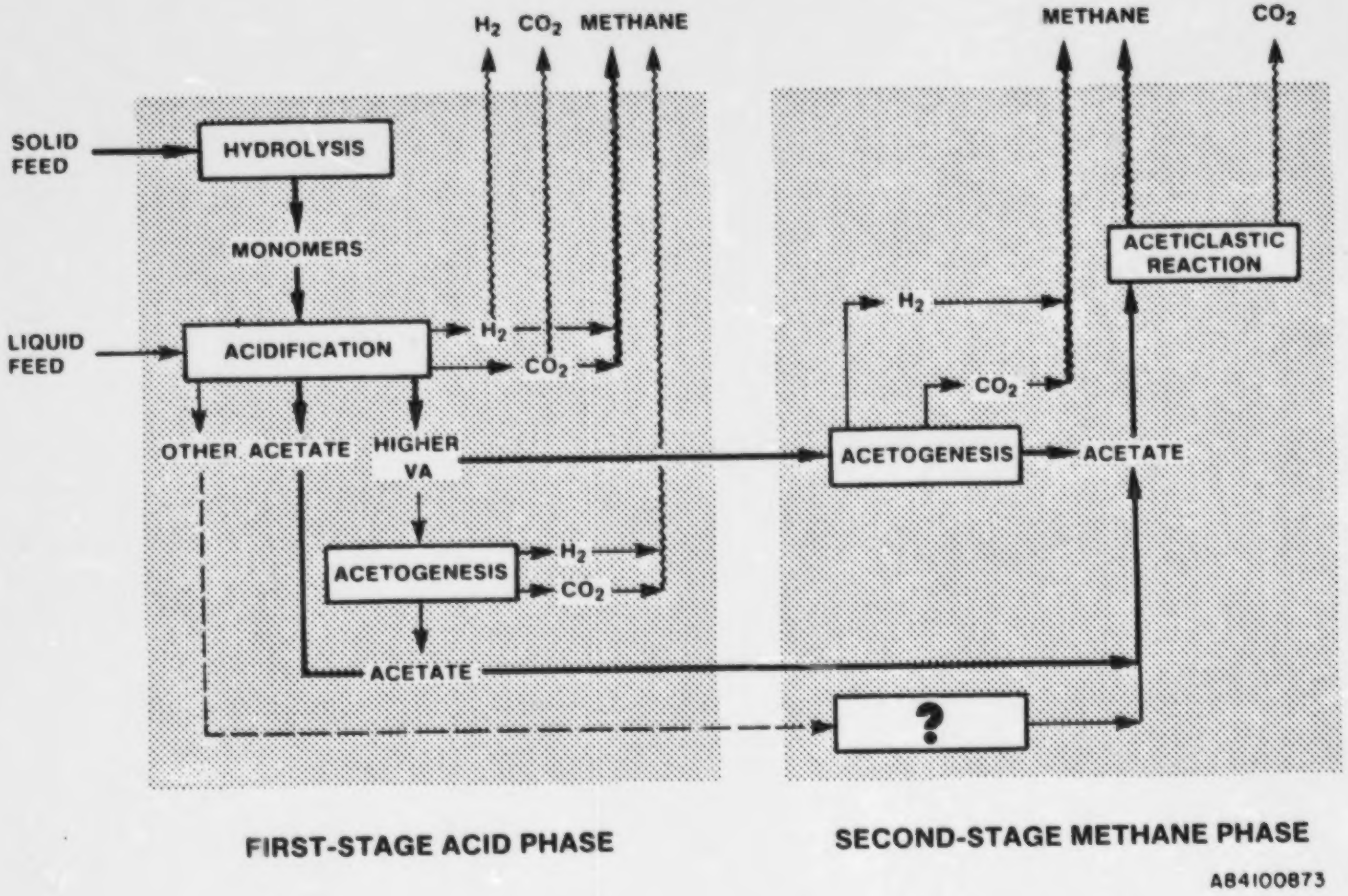
- First, the kinetically dissimilar reaction steps of the overall digestion steps must be optimized in isolated environments or reactor stages because this is not achieved in single-stage mixed-phase digesters, as indicated by the data in Table 1.

- Second, novel digestion reactor designs that provide high substrate and microbial solids retention times (SRT's), must be developed and applied.

TWO-PHASE ANAEROBIC DIGESTION

The Phase-Optimization Concept

There is ample evidence in the literature indicating that the environmental requirements and the growth kinetic characteristics of acidogenic and acetogenic-methanogenic bacteria are very different from each other.[17,19,26-35] Optimization of the above kinetically dissimilar microbial phases in separate digesters has many advantages which include enhanced net energy production, increased process stability, maximized substrate conversion, decreased digester size and plant capital and operating costs, and production of a higher methane-content gas. Phase separation can be achieved by inhibition of methane formers,[36] dialysis separation,[37] or kinetic control.[28] Phase separation by kinetic control of nonmethanogenic and acetogenic-methanogenic bacterial growth by adjustment of HRT and reactor loading rate is the simplest and has been applied by several researchers since it was first demonstrated by Pohland and Ghosh.[19] In the two-stage two-phase approach, hydrolysis and acidogenesis are dominant in the first-stage digester and aceticlastic methanogenesis is the predominant reaction in the second-stage digester (Figure 1). Methane fermentation by carbon dioxide reduction, which is mediated by hydrogenotrophic methanogens and is faster than the aceticlastic reaction and is recognized to be the primary source of methane in anaerobic digestion, is not dominant in the lower-HRT acid-phase digester. Acetogenesis, which is the process of oxidation of higher fatty acids to acetate, is supposedly a slow reaction because of the unfavorable free energy of reaction, and may not be an important conversion step in the first-stage digester. For highly biodegradable liquid substrates which could be rapidly converted by fermentative pathways to acids and molecular hydrogen, acetogenesis may not occur in the first-stage digester and may thus be shifted to the


H2 CO2 METHANE
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SOLID FEED
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LIQUID FEED
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FIRST-STAGE ACID PHASE
SECOND-STAGE METHANE PHASE
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Figure 1. Two-phase anaerobic digestion process concept.

second stage. On the other hand, some acetogenesis may occur in the first-stage digester when it is charged with particulate substrates. Acetogenic conversion could also occur in the first-stage digester when higher HRT's or SRT's are used to promote hydrolysis at a higher efficiency.

Phase Separation with CFCSTR Bioreactors

Commercial high-rate digesters are intermittently or continuously mixed by compressed digester gas, mechanical agitation, or recirculation of the digester contents. The first proposed two-phase digestion process configuration consisted of two conventional CFCSTR reactors operated in series.[19] Anaerobic settlers can be installed in tandem with each digester to permit densification and recycling of settled effluent solids to increase microbial and substrate solids retention times (SRT_m and SRT_s). Figure 2 represents a physical model of the two-phase anaerobic digestion process utilizing complete-mix digesters and anaerobic solid-liquid separators. The purpose of the solid-liquid separator, according to classical concepts, is to produce a concentrated stream of microbial mass and to selectively retain the microbial solids within the digestion system longer than the HRT. In this way, the microbial solids residence time (SRT_m) is higher than the HRT, and the efficiency of the digestion process is enhanced commensurate with the value of the ratio of SRT_m to HRT.

Application of Novel Biodigesters

With few exceptions, solids-liquid separation is difficult with most anaerobic digester effluents, and separate settling of the effluent solids as shown in Figure 2 is not practical. Novel reactor designs which combine solids retention with digestion, and are equivalent in performance to the classical digester-separator combination shown in Figure 2, have been developed and applied successfully during the last two decades. In these digesters the microbial and substrate solids residence times are considerably higher than the HRT, and these reactor characteristics provide the following dual benefits:

- For a given HRT, the high-SRT novel digester exhibits a higher substrate conversion efficiency than that of a complete-mix digester.
- The critical or wash-out HRT of a novel digester is considerably lower than that of a complete-mix digester, which means that the former digester can provide a selected conversion efficiency at a much lower HRT than that of the latter.

The benefits of acidogenic conversion of soluble and particulate substrates in high-SRT digesters are illustrated in Figures 3 and 4. In these examples, the novel digester was assumed to have a microbial or a substrate solids residence time that was double the HRT. The growth kinetic constants (maximum specific growth rate, μ, and saturation constant, K, for the soluble carbohydrate and the sewage sludge volatile solids substrates are shown on Figures 3 and 4; these kinetic constants and the biokinetic models on which these curves of Figures 3 and 4 were based on work presented in an earlier paper.[38] Inspection of Figure 3 shows that a high-SRT novel digester can

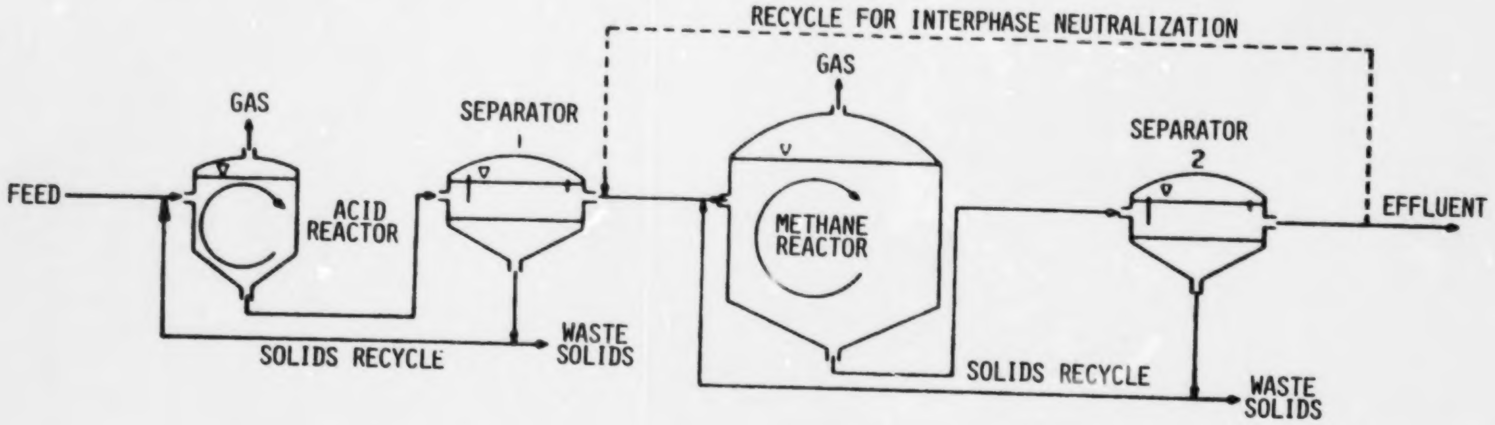
RECYCLE FOR INTERPHASE NEUTRALIZATION
GAS
SEPARATOR
1
GAS
SEPARATOR
2
FEED
ACID
REACTOR
METHANE
REACTOR
EFFLUENT
WASTE
SOLIDS
SOLIDS RECYCLE
SOLIDS RECYCLE
WASTE
SOLIDS

Figure 2. Physical model of the two-phase anaerobic digestion process.

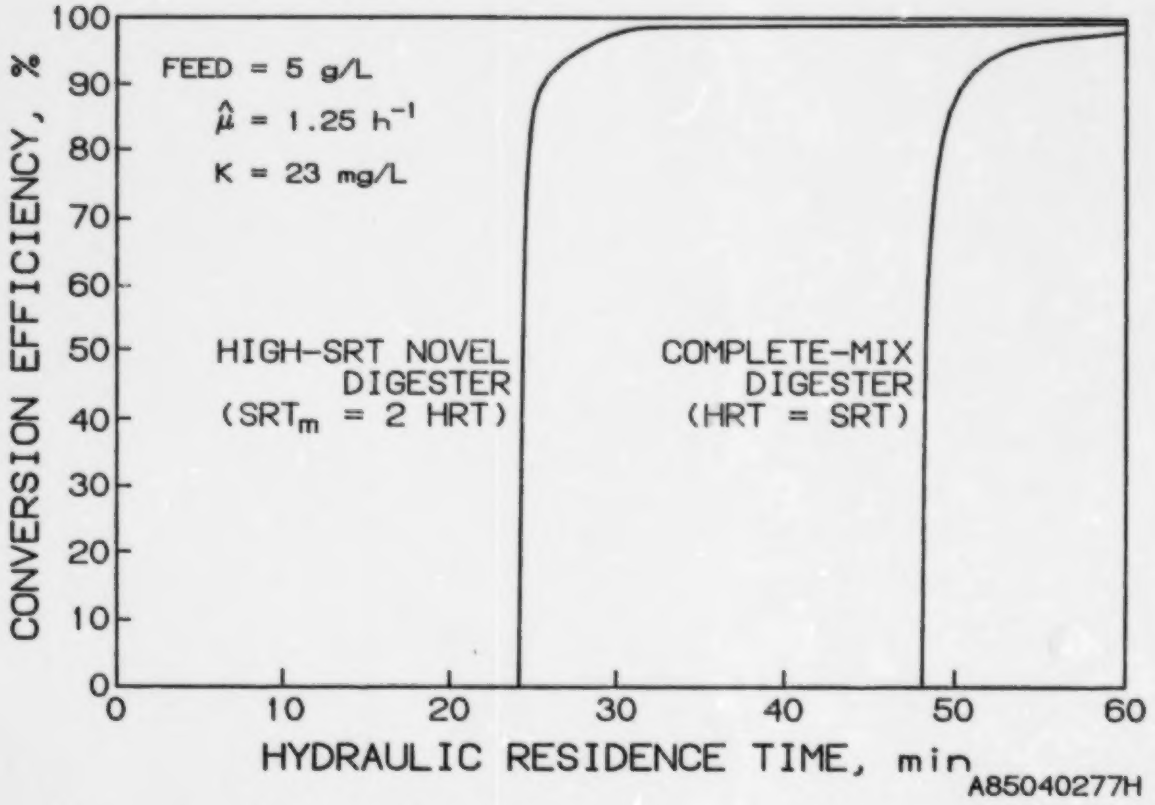

Figure 3. Efficiency of acidogenic conversion of a soluble carbohydrate substrate (glucose) by complete-mix and high-SRT novel digesters.

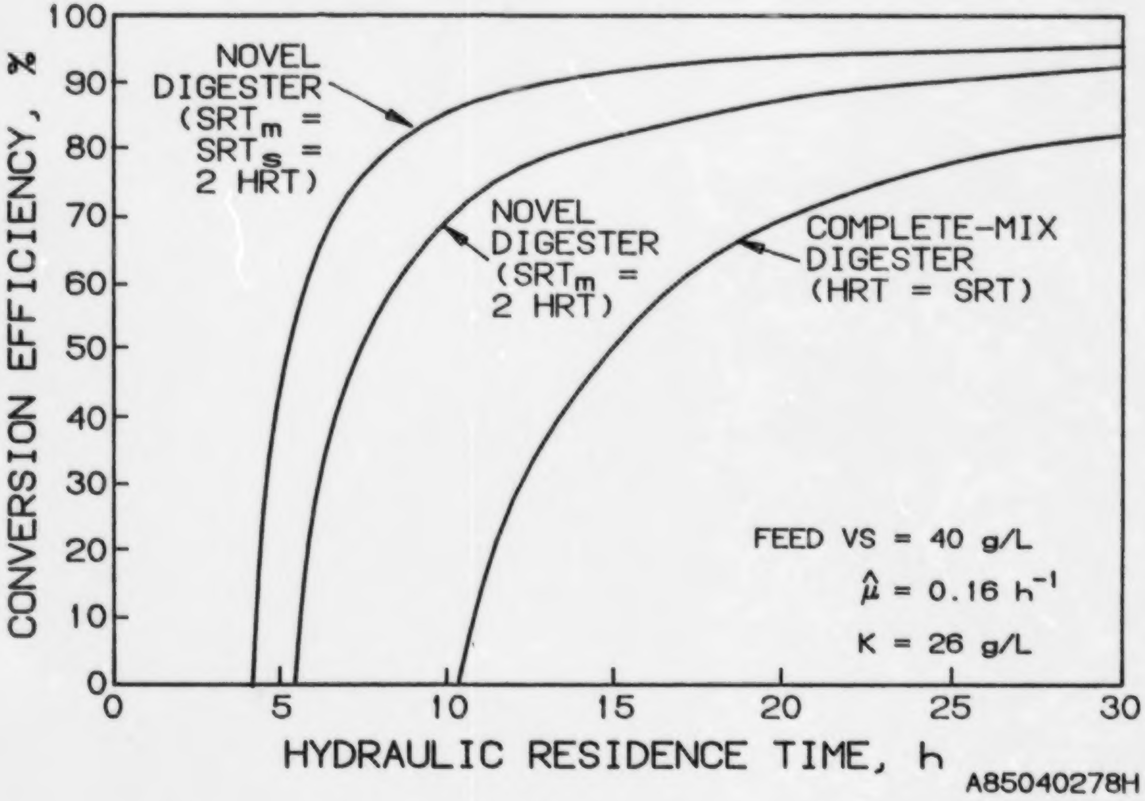

Figure 4. Efficiency of acidogenic conversion of municipal sludge volatile solids by complete-mix and high-SRT novel digesters.

exhibit the same conversion efficiency as that of a complete-mix reactor, but at one-half the HRT of the latter digester; this superior performance of the novel digester in converting the soluble feed is due to enhanced retention of the microbial solids. Novel digesters would exhibit similar improvement in performance when charged with particulate feeds because of microbial solids retention. However, like microbial solids, substrate solids are also retained longer than the HRT, and an additional increase in efficiency over that effected by microbial solids retention alone is realized by use of the high-SRT novel digesters. Thus, as indicated in Figure 4, a complete-mix conventional digester requires an HRT of about 28 hours to exhibit an 80% conversion efficiency with sludge VS; a novel digester in which the SRT_m and SRT_s are twice the HRT, the same conversion efficiency can be achieved at an HRT of about 8 hours.

High SRT novel digesters that utilize suspended-growth cultures and combine digestion with solids retention within the same vessel, and are primarily suitable for soluble feeds, include the Dorr-Oliver Claridigester, the Tower digester developed by the University of Sydney, Australia, and the Baffle-Flow digester developed by Prof. McCarty of Stanford University. In still other novel reactor designs, microbial solids are retained by immobilizing the cells on to static or moving media. The UASB Biothane and the Dorr-Oliver Anitron digesters and the biodisc reactor are examples of cell retention by immobilization on moving media (e.g., "granules" for UASB and sand for the Anitron digesters). By comparison, the Celrobic and the Bacardi downflow anaerobic filters retain solids by fixation of cells on static media used to pack the digestion vessel. A system that is of hybrid design and utilizes both the suspended-growth and the fixed-film cultures has also been applied successfully in laboratory scale.[24] A more detailed discussion on the design and operating characteristics of the above "novel" digesters can be found in a recent publication.[24]

All the novel digesters discussed above are suitable for low suspended-solids content soluble feeds. Novel digesters that are expected to provide high SRT's and are suitable for particulate feeds include the Pfulg-Enerbio digester,[24] the ENEA plug-flow digester, and the upflow digester with solid deflectors.[23]

Advantages of Two-Phase Digestion Based on Kinetic Considerations

The application of kinetic control to separate the acid-forming and methane-forming phases of anaerobic digestion of soluble substrates was first demonstrated by Pohland and Ghosh,[19,26] and later by Ghosh et al,[28] Ghosh and Klass,[38,39] Heertjes and van der Meer,[40] Smith et al.,[41] Cohen et al.,[42] and Ghosh and Henry.[34]

Pohland and Ghosh,[19,26] and Ghosh and Klass[38] studied the kinetic characteristics of acid-forming and methane-forming organisms derived from a digested sewage sludge inoculum and reported the kinetic constants for the acidogenic and methanogenic mixed cultures (Table 2). Kinetic rate constants for acidogenic and methanogenic bacteria were assumed based on previous experiences and results for two-phase and combined-phase systems fed with 70 g/L (of volatile solids) in sewage sludge. These assumed constants were

TABLE 2. ESTIMATED KINETIC CONSTANTS FOR MESOPHILIC (37°C) ACIDOGENIC AND METHANOGENIC CULTURES GROWN ON SOLUBLE AND PARTICULATE SUBSTRATES

	Acid formers grown on glucose		Acid formers grown on sewage sludge	Methane formers	
Kinetic constant	Batch cultures	Continuous culture	Continuous culture	Mixed volatile acids substrates from glucose (Continuous culture)	Acetate substrate (semicontinuous culture)
Maximum specific growth rate, (μ), day^{-1}	7.2	30.0	3.84	3.4	0.49
Saturation constant, (K), mg/L	400 as glucose	23 as glucose	26.0 as VS	600 as acetate	4200 as acetate
Cell yield coefficient	0.15	0.17	0.4	—	0.28

used to generate theoretical performance curves for acid-, methane-, and combined-phase digesters indicated in Figures 5 through 7.

Examination of the data in Table 2 shows that the values of the kinetic constants depend to a significant extent on the nature of the substrate and the culturing mode. It is reasonable to expect, therefore, that considerable improvement in individual culture kinetics may be achieved by optimizing the growth environment for each class of digesting bacteria. Thus, by separate culturing of the acidogenic and methanogenic organisms, it is possible to maximize volatile solids conversion (hydrolysis) and volatile acids production (acidification) rates in the acid-phase digester, as shown in Figure 5. For the operating conditions indicated in Figure 5, volatile acids (acetate) conversion to methane (methanogenesis) cannot occur in the acid digester below a critical HRT of about 2.3 days, and the volatile acids conversion rate is lower than its production rate at higher HRT's. Conversely, volatile acids conversion rate (methanogenesis) is maximized in the separate methane digester, and is much higher than volatile acids production rates at all HRT's (Figure 6); these characteristics of the separate methane digester lead to increased process stability and insurance against unbalanced acidogenic-methanogenic fermentation, which could occur in a single-stage digestion process at low HRT's as shown in Figure 7.

In contrast to the operating characteristics of the separate methane digester of a two-phase digestion process, volatile acids production rate is higher than volatile acids conversion rate in a single-stage CFCSTR conventional digester unless it is operated at a high HRT of about 22 days or higher for the example process depicted in Figure 7. Single-stage digester operation at lower HRT's means 1) unbalanced acidogenic-methanogenic fermentation and the consequent accumulation of volatile acids which in turn leads to inhibition of the methane formers, and 2) maintenance of the hydrolytic and acidogenic bacteria at low growth rates and in the stationary or endogenous growth phases which could be significantly deleterious to the overall conversion process.

A comparison of the theoretical performances of the two-phase and single-stage CFCSTR processes whose operating characteristics are depicted in Figures 5 through 7 shows that the two-phase process could be operated at a much higher loading rate and a much lower HRT than those of the single-stage process, and yet would provide a 79% increase in methane production rate with both processes exhibiting about the same methane yield (Table 3).

In developing the above theoretical treatment, kinetic models applying to acetate-utilizing methanogens, which are predominant and rate-limiting in sludge-fed methane digesters, were utilized. In addition, substrate inhibition of methanogenesis was not considered since it was assumed that residual volatile acids concentration in the single-stage and the methane digester of the two-phase system would be maintained at non-inhibitory levels by appropriate selection of digester HRT and the loading rate. Kinetic models utilized to develop the theoretical basis for superior performance of the two-phase anaerobic digestion relative to the single-stage process, were reported in earlier publications.[26,28,29,38,43] Two-phase process performance observed

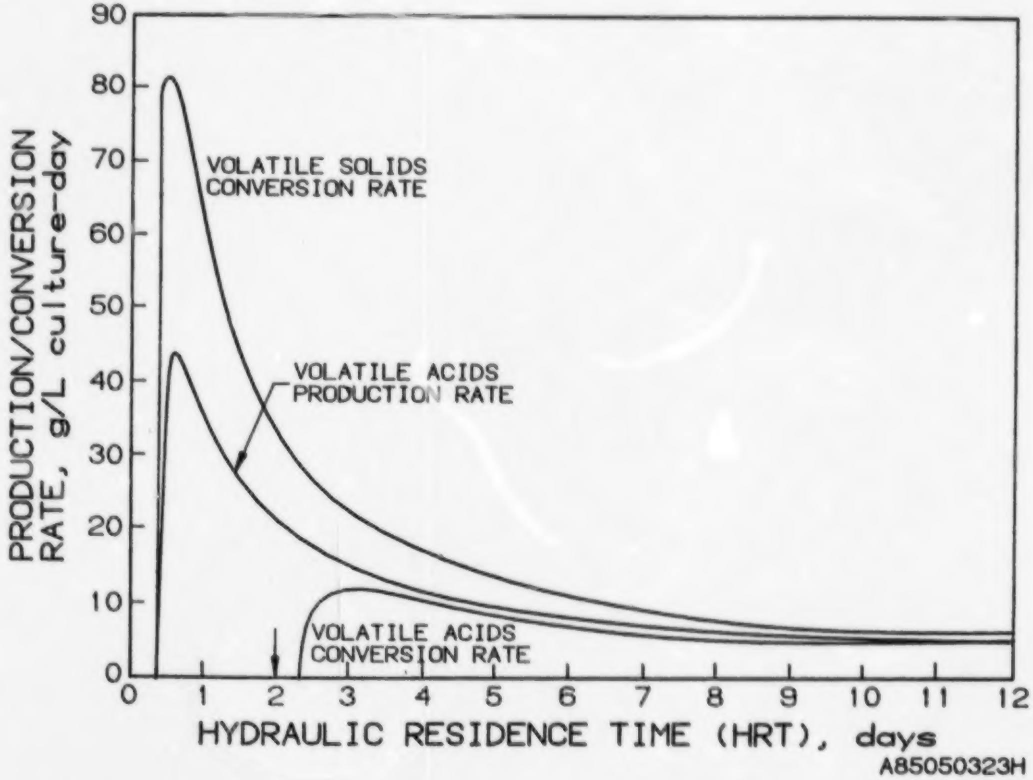

Figure 5. Operating characteristic of a complete-mix acid-phase digester charged with sewage sludge having a volatile solids concentration of 70 g/L (kinetic constants assumed were $\hat{\mu} = 3.84\ \text{day}^{-1}$ and $K = 26$ g/L for acid formers, and $\hat{\mu} = 0.49\ \text{day}^{-1}$ and $K = 4.2$ g/L for methane formers).

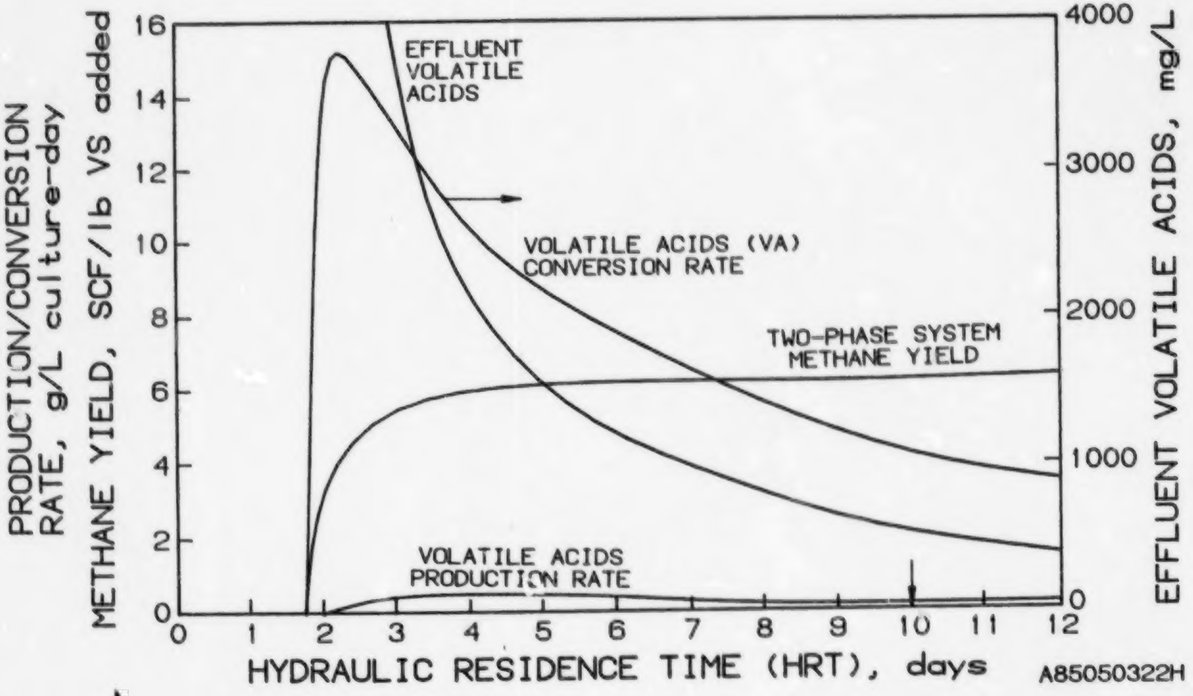

Figure 6. Operating characteristics of a complete-mix methane digester charged with effluents from an acid-phase digester which is operated with a sludge volatile solids concentration of 70 g/L (see Figure 4.5) at an HRT of 2 days (kinetic constants assumed were $\hat{\mu} = 3.84$ day^{-1} and K = 26 g/L for acid formers, and $\mu = 0.60$ day^{-1} and K = 3 g/L for methane formers).

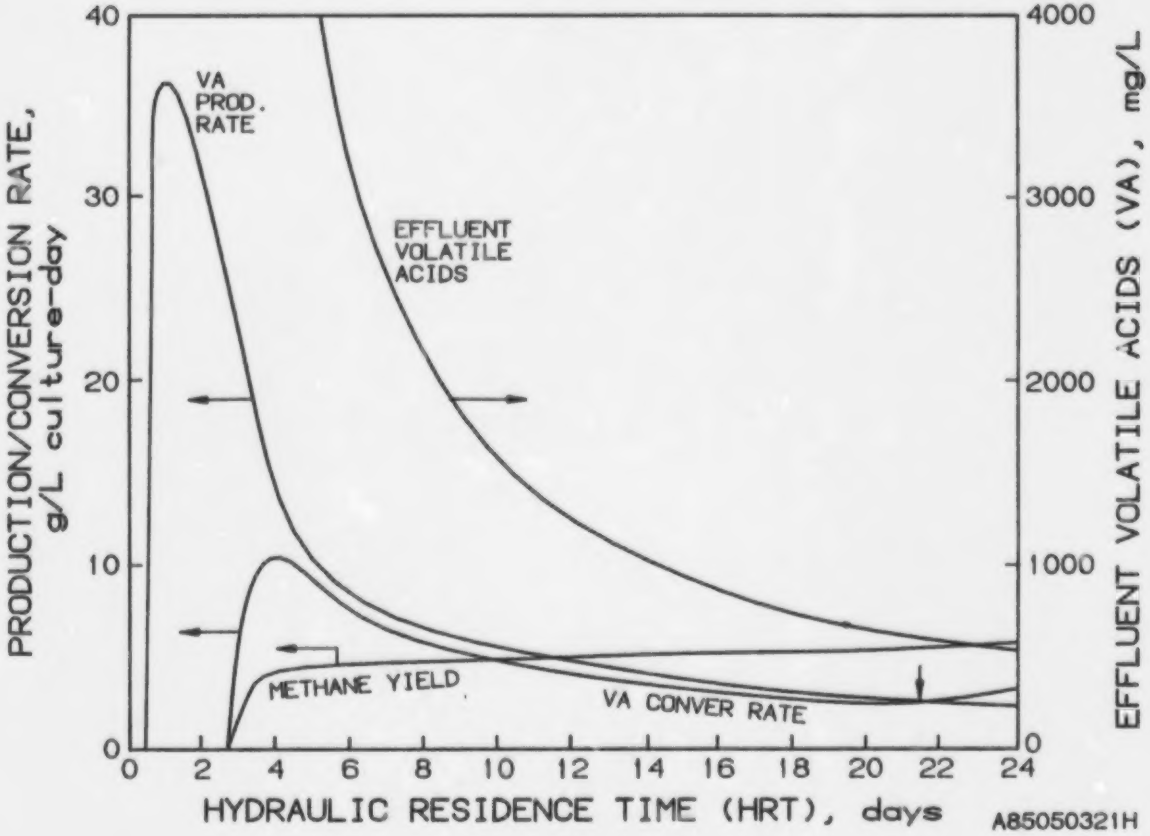

Figure 7. Operating characteristics of a single-stage complete-mix conventional digester charged with sewage sludge having a volatile solids concentration of 70 g/L (kinetic constants assumed were μ 3.84 day^{-1} and K = 36 g/L for acid formers, and μ = 0.49 day^{-1} and K = 6 g/L for methane formers).

TABLE 3. COMPARISON OF THEORETICAL PERFORMANCES OF CFCSTR SINGLE-STAGE AND TWO-PHASE DIGESTION OF SEWAGE SLUDGE*

	CFCSTR Two-phase digestion			CFCSTR single-stage
	Acid-phase	Methane-phase	System	
Sludge VS concentration, g/L	70	--	70	70
HRT, days	2	10	12	22
Loading rate, kg VS/m^3-day	35.0	7.0	5.8	3.2
Methane production rate, SCM/m^3-day	0	3.0	2.5	1.4
Methane yield, SCM/kg VS added	0	0.385	0.385	0.365
Residual VA in methane digester, mg/L	--	600	--	610

* Kinetic constants assumed in developing the performances are reported in Figures 5 through 7. It was assumed that gas from the single-stage digester has a methane content of 60 mol % and that gas from the acid digester has 70 mol % methane. It was further assumed that 90% of the volatile acids is converted to gas in the two-phase system compared with 85% for the single-stage digester.

in this project were generally in agreement with the type of projections that can be made from these models and are presented in Table 3.

The performance of a two-phase system is expected to be superior to that of a single-stage system irrespective of the reactor type used as the digestion vessel. The theoretical development presented in the foregoing paragraphs was based on the application of CFCSTR digesters. But similar development is possible for other novel high-SRT bioreactors. For example, it could be shown that a two-phase system consisting of a UASB acid digester and a UASB methane digester exhibits superior performance relative to a single-stage UASB system.

Historical Development of the Two-Phase Concept

Babbit and Baumann[22] were probably the first to suggest that separation of the anaerobic digestion process into "two or more stages" may have the advantage of overcoming the inhibitory effects of the intermediate products (for example, volatile acids) in the early stages (hydrolysis and acidification) of digestion. The first experimental work on two-phase digestion was conducted by Hammer and Borchardt,[44] and Schaumburgh and Kirsch.[36] These researchers attempted to separate the acid and methane phases by dialysis membranes, selected inactivation of the acid- or methane-formers by appropriate inhibitors.[45,46] The complexity and operational difficulties of these techniques with acidogenic and methanogenic organisms, however, make them unattractive and impractical. Since the cited work, little interest has been expressed in these methods for separating the microbial phases of anaerobic digestion.

Phase Separation by Kinetic Control

A much simpler and more practical technique to separate and maintain dominant microbial phases is that studied by Ghosh and Pohland.[45] This technique, termed *kinetic control*, relies on the principles of population dynamics and enrichment of the acidogenic and methanogenic phases in separate digesters by simple operational adjustment of the reactor dilution rate or the organic loading rate and cell mass recycle ratios. The objectives of these adjustments are to exceed the maximum specific growth rate of the acetate-utilizing methane formers by the allowable growth rate in the first reactor (acid digester), and to promote maximized conversion of the biodegradable substrates in the first phase to volatile acids and other intermediates acceptable to the acetogenic bacteria and the methane formers. Considerable work has been done to accomplish phase separation by kinetic control since Pohland and Ghosh reported the results of their phase separation studies in 1971. Various configurations of two-phase digestion have been developed for the digestion of liquid, "semi-solid," and "solid" substrates which are briefly described in the following sections.

Two-Phase Digestion of Soluble Substrates

The application of kinetic control to separate the acidogenic and methanogenic phases of anaerobic fermentation of soluble substrates was first demonstrated by Pohland and Ghosh,[19,26] and later by Heertjes and van der Meer,[47] Smith et al.,[41] Cohen et al.,[42] and others. Ghosh and Pohland[45] also presented kinetic models describing the velocities of production as well as the concentrations and yields of product acids and gases from two-phase digestion of soluble substrates. A comparative study of the kinetic characteristics of the acidogenic and methanogenic organisms was also presented.

Various reactor designs, differing from the completely mixed digesters used by Pohland and Ghosh, were studied by other researchers. Cohen et al.[42] experimented with a two-phase system consisting of a completely mixed acid-phase reactor and a plug-flow type upflow methane digester with a built-in settler to conduct anaerobic digestion of glucose. The cell yield coefficient in the acid-phase digester at 30°C was 0.11, compared with a yield coefficient of 0.17 at 37°C reported by Ghosh and Pohland.[29,45] Ethanol, acetate, propionate, butyrate, formate, lactate, carbon dioxide and hydrogen were the main products of acidogenesis. Butyrate was produced in the largest concentrations, followed by acetate. The acidogenic reaction products were gasified in the upflow methane digester to produce head gases having 84.3 mol % methane and 15.7 mol % carbon dioxide.

Ghosh[47] and Ghosh and Henry[34] operated a CFCSTR acid-phase and an upflow packed-bed methane digester with real soft-drink bottling waste, and demonstrated that a two-phase digestion process could be operated at about seven times the loading rate and one-half the HRT of the conventional process and still obtain the same methane production as, and a slightly higher COD reduction than, the conventional process (Table 4). An important advantage of the two-phase process was that gases from the methane phase had a significantly higher methane content than those of the conventional digester. It was projected that two-phase operation would allow the total digester volume (and associated capital and operating costs) to be reduced by 67% and the net energy production to be increased by more than 73% relative to those of the conventional process. Also, while the conventional high-rate digester failed at an HRT of 10 days and a feed COD concentration of 26,000 mg/L, the two-phase process exhibited stable and efficient performance at a system HRT of 7.4 days and a feed COD concentration up to 45,000 mg/L.

The two-phase digestion process has been proven in both pilot- and full-scale operation at overall loading rates up to 12 kg COD/m^3-day and HRT's down to 13 hours, affording the same or higher methane yields as achieved at one-tenth the loading rate and ten times the HRT needed for stable operation of a single-stage high-rate digester.[48,49] The commercial process (known as the Anodek process in Europe) utilized a CSTR acid-phase digester operated in tandem with a UASB methane digester.[48,49]

Heertjes and van der Meer[40] also conducted two-phase digestion of saccharose and sodium acetate in an upflow digester with an internal settler built at the top (effluent end) of this digester. High conversion efficiencies were obtained at 3- to 6-hour residence times and a relatively low loading (1.92 kg TOC/m^3-day). A two-reactor two-phase system exhibited increased stability at higher loadings up to 11.84 kg TOC/m^3-day.

Smith _et al_.[41] operated a packed-bed mesophilic (37°C) upflow methane digester ("anaerobic filter") with solids-free acidic substrates derived from animal wastes. Satisfactory acid-phase digestion could not be developed with this waste. Methane digester gas production rates from 0.24 to a high value of 2.77 volume/digester volume-day were observed at hydraulic retention times of 1.1 to 40.5 days.

Pipyn _et al_.[30] investigated anaerobic digestion of distillery wastewaters (~10,000 mg/L COD) in a two-phase pilot plant consisting of a 36-m^3 CFCSTR acid-phase digester and a 5-m^3 upflow methane-phase digester. The acid-phase was operated at 42°±2°C at an HRT of 16 to 72 hours, while the methane-phase digester was maintained at 39°±2°C and an HRT of 14 hours. Overall COD and BOD reductions of 84% and 92% were obtained. The methane digester gases had a methane content of 75±3 mol %.

TABLE 4. HIGH-RATE AND TWO-PHASE MESOPHILIC (35°C) DIGESTION OF SOFT-DRINK BOTTLING WASTE

	Conventional high rate	Two-phase		
		Acid phase	Methane phase	Overall
Loading, kg VS/m^3-day	0.64	16.0	6.4*	4.8
HRT, days	15	2.2	5.2*	7.4
Methane yield, SCM/kg VS added	0.64	0.06	0.59	0.61
Methane content, mol %	61.1	0.2	70.5	63.1
Gas production rate, SCM/m^3-day	0.4	1.03	3.68	2.90
Digestion efficiencies, %				
VS reduction	72	--	--	64
COD reduction	84	--	--	96
Digester volume for 9090 kg/day TS load, 1000 m^3	5.6	0.6	1.2	1.8
Net energy production				
10^6 kcal/day	11.7	--	--	20.2
Percent of total production	37	--	--	62

* Loading and HRT of the upflow filter methane digester were calculated on the basis of the gross volume of the packed bed.

Two-Phase Digestion of Semi-Solid Substrates

Ghosh *et al.*[28] and Ghosh and Klass[38] first demonstrated the feasibility of separating the acid and methane phases of anaerobic digestion of a particulate feed (activated sludge) by kinetic control. Satisfactory acid-phase digestion occurred at hydraulic retention times of 10 to 24 hours and high loadings of 32 to 80 kg VS/m^3-day. Acidogenesis occurred at an oxidation-reduction potential (E_c) of -240 mV and a pH of 5.7, compared to -400 mV and 7.0 for methane formers. Kinetic constants were determined for both phases of activated sludge digestion. Methanogenesis was the rate-controlling step of the overall digestion process. The methane digester gases contained 70 mol % methane. One important finding from this work was that high reactor loadings are required to maximize acid production rate per unit reactor volume and to minimize acid digester retention time.[38,50] Two-phase mesophilic digestion of 1.7 to 2.5 wt % VS-content Chicago activated sludge exhibited an average methane yield of 0.27 SCM/kg VS added and a VS reduction of 40%[28] at an overall HRT of 6.9 to 7.7 days compared with 0.22 SCM/kg VS added and 34% observed during conventional digestion of this sludge at an HRT of 14 days. The methane content of conventional digester gases was 60 mol % compared with 70 mol % in the head gases of the methane-phase digester.

Eastman and Ferguson[27] conducted acid-phase digestion of primary sewage sludge at HRT's of 9 to 72 hours, and concluded that hydrolysis of the solid sludge particles was the rate-limiting step of the overall acidogenic phase. Lipids were not biodegraded, and 50% of the non-lipid COD of primary sludge was solubilized. Acidogenic sludge was difficult to settle. Hydrogen evolution occurred at the minimum detention time of 9 hours. Volatile acid production and distribution of acid species in the effluent appeared to be influenced by the reactor pH. Brown[51] indicated that hydrolysis of particulate substrate was favored at an acidic pH (pH 6), and methane fermentation of the acid-digestion products was better at an alkaline pH (pH 7.5). Detailed investigation of the pH effect, however, was not conducted to delineate the pH optima. The methane digester gases contained 80 mol percent methane.

Norrman and Frostell[52] conducted mesophilic (33°C) two-phase digestion of a semi-solid synthetic feed (blended dog food) in a laboratory system comprised of a completely mixed acid-phase digester and a packed-bed upflow methane digester. The acid digester was followed by a 500-mL gravity settler, the supernatant from which was fed to the packed-bed methane digester. Acid digester pH was low (pH 4). Solid-liquid separation was a problem with the acid-digester effluent. The overall system was operated at HRT's of 2.7 to 12.1 days and low loadings of 0.42 to 2.24 kg VS/m^3-day. A long starting time was required for the anaerobic filter. The methane digester gases contained 65 to 80 mol % methane. Like Norrman and Frostell, Therkelsen and Carlson[53] also investigated the two-phase digestion characteristics of dog food, but at a thermophilic temperature of 50°C. The performances of completely mixed and plug flow acid digesters were compared. Surprisingly, lactate was the major acidic product. The pH of the acid digester was also low (pH 4) and grease and organic nitrogen were not reduced significantly. One interesting observation was that acid production in a plug-flow acid digester was much higher than that in the complete-mix reactor. At the test loadings (5.9 to 9.9 kg VS/m^3-day) and HRT's (4.3 to 7.5 days), two-phase thermophilic digestion of dog food was slightly better than thermophilic conventional digestion.

Keenan[54] conducted two-phase digestion of simulated solid waste (Purina Dog Chow) at 22° and 48°C. The acid-phase digester had relatively long HRT's of 4.5 and 6 days; the methane digester had a HRT of 10 days. Acid digester gases contained mainly CO_2 and a small amount of hydrogen. Gases from the methane digester had 80 mol percent methane. The acid digester effluent had 13,000 to 14,000 mg/L of volatile acids. There was no significant difference in acid conversion efficiencies at 22° and 48°C. The two-phase process exhibited higher stability than the conventional mixed-phase high-rate process.

In contrast to the two-reactor systems studied by most researchers, Johnson[55] found evidence of separation of the acidogenic and methanogenic phases during anaerobic fermentation of pig excrement and biomass leachate in a four-stage system. The two-phase multi-stage process was superior to conventional high-rate digestion.

Two-Phase Digestion of Semi-Solid Feeds With Novel Upflow Bioreactors

A review of the literature on digestion of such particulate feeds as sewage sludge, municipal solid waste, manure, and various biomass species showed that mesophilic digestion of these feeds is generally conducted at an HRT higher than 10 days and at loading rates lower than 3.2 kg VS/m^3-day.[23,56] Best methane yields and methane production rates are less than 0.42 std m^3/kg VS added and 1.9 vol/culture vol-day, respectively. These performances were significantly exceeded during digestion of sewage sludge in an advanced two-phase system comprised of custom-designed and unmixed upflow digesters operated in series to optimize the liquefying-acidification and acetogenesis-methanation reactions.[23] The system was operated in a continuous mode for about 16 months and exhibited a progressively increasing methane yield at HRT's of less than 6 days. With continuing culture enrichment and improvements in reactor design, the methane yield increased from 0.31 to 0.43 SCM/kg VS added, and then to 0.48 SCM/kg VS added (Table 5). This methane yield was about 77% of the theoretical methane yield achievable with this sewage sludge and is the highest methane yield reported for sludge at this HRT. Operation of the novel process configuration was very stable and superior to that of conventional single-stage digestion in terms of methane yield, gas production rate, and net energy production. Considerations of the volatile suspended solids (VSS) and particulate (or solid-phase) COD inputs to and outputs from the acid- and methane-phase digesters showed a liquefaction efficiency between 46 and 55% in the first-stage digester compared with a liquefaction efficiency between 0 and 10% only for the methane digester.[23] Acetogenesis and methanogenesis predominated in the second-stage digester. Whereas little methane fermentation occurred in the acid-phase digester at an HRT of 1.1 days, methane production increased 20-fold when the first-stage HRT was increased to 1.3 days; acetate was the major volatile acid at the lower HRT, but propionate predominated at the higher HRT.

As shown in Table 6, a 91 metric ton/day hypothetical two-phase upflow digestion plant requires about 60% of the digester volume needed for a conventional single-stage system, and exhibits a VS reduction three times that of the latter process. These performances translate to substantial savings in capital and operating costs. The single-stage conventional process is a net energy consumer for a low-HRT operation. By comparison, about 83% of the digester methane is available as surplus bio-fuel if a two-phase upflow digestion process is used.

Two-Phase Digestion of Solid Feeds

The two-phase digestion systems described above are suitable for semi-solid feeds; the above process configurations may not be applied to gasify and stabilize solid feeds unless they are diluted to form slurries. This dilution approach, although commonly employed, is not attractive for many reasons. A

TABLE 5. PERFORMANCE OF AN ADVANCED TWO-PHASE UPFLOW MESOPHILIC (35°C) DIGESTION SYSTEM AT AN HRT OF 5.9 DAYS (1.3 Days for Acid Phase and 4.6 Days for Methane Phase) WITH A 5.8 wt % TS-CONTENT FEED

Operating conditions/performance	Acid phase	Methane phase	System
Run duration, number of HRT's	35	10	8
Loading rate, kg VS/m^3-day	28.8	7.8	6.2
Gas production			
Methane yield, SCM/kg VS added	0.06	0.42	0.48
Methane content, mol %	59.2	70.1	68.4
Methane production rate, SCM/m^3-day	1.77	3.27	2.96
Effluent characteristics			
pH	6.6	7.2	7.2
Volatile acids, mg/L			
Acetic	643	77	77
Propionic	2251	48	48
Isobutyric	123	0	0
n-Butyric	141	0	0
Isovaleric	266	0	0
n-Valeric	79	0	0
Caproic	0	0	0
Total as acetic	2827	118	118
Ethanol, mg/L	0	0	0

special process configuration — leach-bed two-phase digestion — is more suitable for "dry" or high-solids-content feeds, is simpler than slurry-phase digestion, and is conducted without dilution of the feed, without mixing, and even under ambient conditions. Leach-bed solid-phase anaerobic fermentation is particularly attractive for such low-moisture organic feeds as municipal and industrial solid wastes, sludge cakes, manure, agricultural and forestry residues, farm wastes, and other similar organic biomass and wastes.

The leach-bed two-phase digestion process overcomes the difficulties of the so-called dry digestion by inducing rapid bio-leaching of the solid feed by application of an acidogenic culture, and promoting continued and accelerated liquefaction and acidification of the bed by recirculation of the reactivated culture (Figure 8). This fermentation approach employs active control of all phases of the overall digestion process. Liquefaction products from the acidogenic leach-bed are moved to an acid-recovery process or are diverted to a separate methane-phase digester for gasification of the volatile acids with recycling of the methane-phase effluent to the leach-bed to

TABLE 6. COMPARISON OF HYPOTHETICAL CONVENTIONAL AND TWO-PHASE UPFLOW MESOPHILIC (35°C) DIGESTION SYSTEMS TO STABILIZE AND GASIFY 91 METRIC TONS/DAY (Dry Solids Basis) OF SLUDGE AT AN HRT OF 5.5 DAYS

	Conventional	Two-stage upflow
Operation and performance		
Feed VS, wt %	2.2	3.7
Loading rate, kg VS/m^3-day	4.0	6.6
Methane yield, SCM/kg VS added	0.13	0.48
Methane production rate, SCM/m^3 vol-day	0.5	2.8
VS reduction to gas, %	24	75
Gross methane production, 10^3 std m^3/day	6.8	26.6
Estimated operating energy requirement, 10^6 kcal/day		
Feed sludge heating	61.2	38.6
Mixing	1.5	0
Pumping	0.5	0.8
Heating, ventilation, lighting, other	2.0	1.3
Total	65.2	40.7
Net energy production, 10^6 kcal/day	4.8	196.6
Digester volume, 1000 m^3	13.6	8.3

conserve the nutrients indigenous to the solid substrate and thus to eliminate or reduce the need for external nutrient addition.

The leach-bed two-phase digestion process is superior to the traditional slurry-culture single-stage digestion process because —

- It is able to handle "dry" or high-solids-containing feeds.
- A minimum of feed processing (e.g., shredding, grinding and separation) and feed pretreatment (e.g., chemical or enzymatic) are necessary.
- Feed slurrification is not necessary.
- Intensive mechanical mixing is not required.
- Addition of external nutrients is eliminated or minimized.
- The process can be applied for in-situ bioconversion of waste deposits (e.g., landfills) and the ultimate disposal of the final residues.

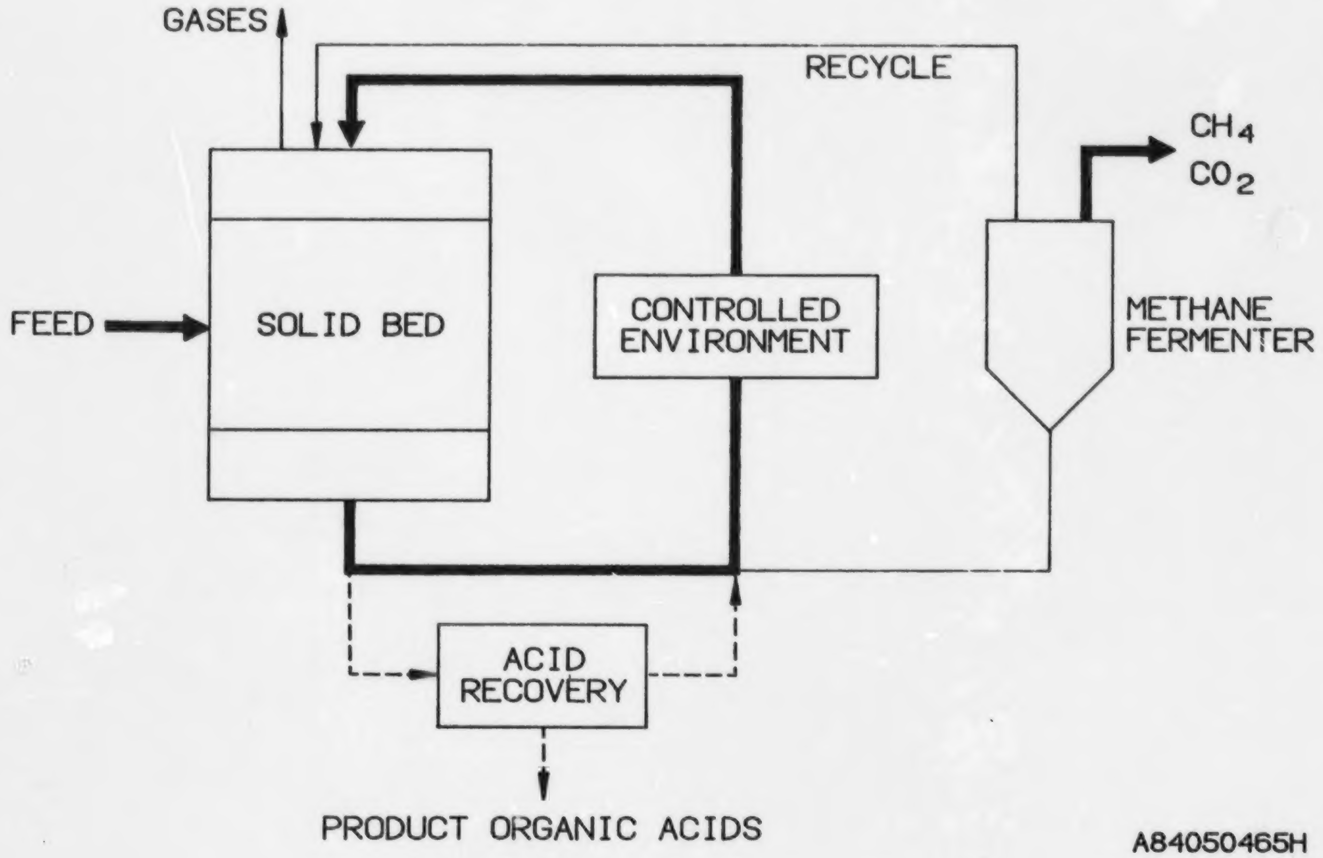
GASES
RECYCLE
CH_4
CO_2
FEED
SOLID BED
CONTROLLED ENVIRONMENT
METHANE FERMENTER
ACID RECOVERY
PRODUCT ORGANIC ACIDS
A84050465H

Figure 8. Leach-bed two-phase anaerobic digestion.

- Fermentation can be conducted in simple containment vessels.
- There are fewer fermenter volume and energy requirements, compared with the conventional dilute-slurry fermentation processes.

Investigation with a small pilot-scale system consisting of a 126-L refuse-derived feed (RDF)-filled mesophilic (35°C) leach bed and a mesophilic 12.5-L methane-phase anaerobic filter exhibited a volatile acids yield of 23% (0.23 g acids/g VS) and a methane yield of 0.31 SCM/kg VS added, indicating complete conversion of the biodegradable fraction of refuse.[56] For batch operation, gasification was virtually completed in about three months. The leach-bed two-phase digestion process can be operated in the sequential-batch, fed-batch, or the semi-continuous fermentation mode. Leach-bed two-phase digestion of municipal landfill is known as the LanFilgas® process.[57]

Benefits of Two-Phase Digestion

Based on data reported in the literature, it appears that two-phase anaerobic fermentation has the potential of fulfilling the need for a short-residence-time and high-efficiency biomass/waste-to-methane conversion process. Acid-phase digestion can be conducted at HRT's as low as 3 to 6 hours for soluble organics and 9 to 48 hours for particulate organic material. The overall two-phase system can be operated at HRT's of 2 to 7 days depending on the feed — a substantial improvement over conventional high-rate digestion conducted at HRT's of about 12 to 20 days.

In addition, two-phase digestion has several other demonstrated and potential benefits as follows:

- The capacity of maintaining an optimum environment for each group of digester organisms, thus optimizing the overall digestion process.
- A substantial reduction in total reactor volume and consequent savings in capital and operating costs.
- Improved mixing in smaller low-residence-time digesters.
- Possibility of auto-mixing by the high-rate of gas production.
- Higher rates of solids stabilization and methane production.
- Much higher methane content (up to 85 mol %) of the final product gas.
- Decreased heat requirements and increased net energy production.
- Suitability for incorporation into existing treatment plants with minimum capital investment.
- Reduction of the nitrogen content of the system effluent by simultaneous liquefaction and denitrification of waste feeds in the acid digester.

- Increased process stability because the sensitive methane bacteria are separated and are not subjected to environmental shocks of sudden acid production and dropping pH.

It is expected that the cost of operating a two-phase digestion system would be significantly lower than that of conventional single-stage digestion because of reduced operating energy requirements, lower residue disposal costs, and lower annualized plant capital cost.

SECTION 5

EXPERIMENTAL PLAN

DIGESTER FEEDS

Since many municipal digesters receive both primary and activated sludges, it was decided that the digestion experiments of this project should be conducted with mixtures of these two sludges to be collected from the Chicago metropolitan area. Another factor that was considered important was the source of the collected sludge. It was reasoned that sludges collected from both large and small sewage treatment plants should be tested. Accordingly, mixed primary-activated, primary, and activated sludges were collected from a suburban and a City-of-Chicago plant of the Metropolitan Sanitary District of Greater Chicago (MSDGC) system and from a plant of a suburban (Downers Grove) sanitary district.

DIGESTION SYSTEMS

Consistent with the objectives of this research, it was necessary to utilize two types of digestion apparatus — two-phase and single-stage digestion systems. Two-phase systems for the fundamental studies comprised CFCSTR acid- and methane-phase digesters, whereas novel upflow and CFCSTR digesters were used to conduct the two-phase runs for the Applied Studies.

The choice of the digester type for the fundamental studies allowed for direct comparison of single-stage and two-phase digestion and a delineation of the effect of the fermentation mode on system performance. Similarly, by cross comparison of two-phase digestion runs with CFCSTR and upflow digesters employed in the fundamental and applied studies, it was possible to assess the effect of reactor design.

DIGESTION RUNS

A total of 27 single-stage and two-phase digestion runs were conducted to accomplish the objectives of Process-Comparison, Parametric-Effects acid-phase, and Applied-Studies digestion runs. Runs were conducted with single-stage (SS) and two-phase (TP) digestion systems at mesophilic (M) and thermophilic (T) temperatures at various HRT's, feed VS concentrations, and pH's, and with and without enzyme treatment of the raw sludge.

A rational numbering system which identifies the digester type, digester HRT, temperature, and culture pH for a particular run was used. The first letters of a run number indicate the digester type (e.g., SS for single-stage, AP for acid-phase, MP for methane phase, TP for two-phase system with CFCSTR digesters, UTP for two-phase system with upflow digesters). The first letters of run number are followed by the digester HRT in days which in turn is followed by the letter(s) M, T, M-M, M-T or T-T denoting single-stage mesophilic (meso) or thermophilic (thermo) temperature, or meso-meso, meso-thermo, or thermo-thermo acid-methane phase temperature combinations of a two-phase system. The digits following the temperature notation indicate the target culture pH to be attained by addition of acid or alkali. A run number with (E) at the end indicates that the feed sludge in this run was treated with cellulose and lipase. As an illustration, Run No. TP3M-M(E) indicates a CFCSTR two-phase run at a system HRT of 3 days with a mesophilic acid-phase digester operated in series with a mesophilic methane digester with enzyme treatment of the feed sludge.

Fundamental Studies

Process-Comparison Studies--

A number of digestion runs were planned as shown in Table 7 to conduct comparative studies of single-stage high-rate and two-phase digestion processes under the same conditions of temperature, HRT, loading rates, and feed VS concentrations. Single-stage digestion runs were planned at fermentation temperatures of 35°C and 55°C and at HRT's of 15, 7, and 3 days with parallel two-phase runs conducted at these same target HRT's. The experimental design thus provided for the pairing of single-stage and two-phase runs based on HRT, and digestion temperature. All digesters of the mesophilic pairs were operated at 35°C. The single-stage and methane-phase (MP) digesters of the thermophilic pairs had a target temperature of 55°C. The acid-phase (AP) digesters of the thermophilic two-phase systems were to have temperatures of 35° or 55°C depending on the system HRT. As shown in Table 7, a mesophilic temperature of 35°C was chosen for the acid digester when the system HRT of the thermophilic two-phase system was high (15 days). For a low system HRT (3 days) both digesters of the thermophilic two-phase process had a target temperature of 55°C. It was reasoned that owing to faster kinetics, a thermophilic temperature could be more appropriate than a mesophilic temperature when the system HRT is decidedly low. For the intermediate HRT of 7 days, two thermophilic two-phase runs were planned, one with the acid-phase digester 35°C and the other at 55°C. Thus, the thermophilic two-phase systems in reality were meso-thermo (35°C AP-55°C MP) or thermo-thermo (55°C AP-55°C MP) processes.

The above experimental design was expected to provide the following information:

- Effects of HRT and temperature on single-stage and two-phase digestion
- Benefits, if any, of two-phase digestion over the single-stage process in terms of gas and methane productions and rates and efficiencies of conversions of VS, total carbohydrate, lipids, and proteins.

TABLE 7. DESIGN OPERATING CONDITIONS FOR PROCESS COMPARISON DIGESTION RUNS CONDUCTED WITH CFCSTR DIGESTERS

Single-stage digestion runs							
Run no.	SS15M	SS7M	SS3M	SS15T	SS7T	--	SS3T
Digester no.	331	331	331	337	331	--	335
Culture temperature, °C	35	35	35	55	55	--	35
HRT, days	15	7	3	15	7	--	3
Organic loading rate, kg VS/m^3-day	2.1	7.1	16.7	2.1	7.1	--	16.7
Feed VS concentration, g/L	31.5	50.0	50.0	31.5	50.0	--	50.0
Two-phase digestion runs							
Run no.	TP15M-M	TP7M-M	TP3M-M	TP15M-M	TP7M-T	TP7T-T	TP3T-T
Digester nos.*	332-333	334-333	334-333	334-337	334-331	335-331	335-331
Culture temperatures,† °C	35-35	35-35	35-35	35-55	35-55	55-55	55-55
HRT, days							
Acid-phase	2	2	0.8	2	2	2	0.8
Methane-phase	13	5	2.2	13	5	5	2.2
System	15	7	3.0	15	7	7	3.0
System organic loading rate, kg VS/m^3-day	2.1	7.1	16.7	2.1	7.1	7.1	16.7
Feed VS concentration, g/L	31.5	50.0	50.0	31.5	50.0	50.0	50.0

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* The first number is that of the acid-phase and the second number is that of the methane-phase.

† The first temperature is that of acid-phase culture and the second temperature is that of the methane-phase culture.

Parametric-Effects Acid-Phase Runs--

A total of 12 CFCSTR acid-phase digestion runs were planned at two temperatures, four pH's and two HRT's (Table 8) to delineate the effects of these operating parameters on acid digestion of sewage sludge. The factorial experimental design follows a three-criteria classification in which a selected variate (e.g., total volatile acid concentration, protein reduction, or gas yield, etc.) is influenced by three treatment variables: digestion temperature, pH, and HRT. The effects of the treatment variables on digester performance as measured by a selected variate (e.g., organic component reduction, volatile acid production) may be evaluated by performing the analysis of variance (ANOVA) test using steady-state values of the variate.

Applied Studies

Advanced Two-Phase Digestion--

Several two-phase experiments were planned with upflow acid and methane digesters maintained at mesophilic and thermophilic temperatures and without and with recycling of the methane digester effluents to the acid phase — it was reasoned that this effluent recycling would moderate the acid digester pH and supply hydrogen utilizing methanogens which, by removing electrons, would enhance hydrolysis and acidification. Screening studies were planned at meso-meso, meso-thermo, and thermo-thermo acid- and methane-phase temperature conditions to be able to select the best reactor temperature combination. It was decided that this advanced two-phase process would be operated at an HRT and pH deemed best from the results of the Fundamental Studies. This experimental design was expected to aid in the selection of optimum operating conditions for the two-phase process. At least one steady-state run was planned at the optimum operating conditions (Table 9).

Enhancement of Sludge Reactivity by Enzyme Treatment--

It is well known that the volumetric rate of anaerobic digestion increases at lower HRT's; however, a decrease in stabilization efficiency is also experienced as the rate of conversion increases. One way to enhance the conversion efficiency at the lower HRT is to increase the reactivities of certain organic components by enzymatic treatment. As mentioned before, cellulase and lipase treatments of the raw sludge were considered to this end in view of the success of this approach in another EPA-sponsored project. The applied studies included a 3-day HRT CFCSTR run that would be conducted with enzyme treatment of the feed sludge (Table 9).

TABLE 8. DESIGN OPERATING CONDITIONS FOR PARAMETRIC-EFFECTS CFCSTR ACID-PHASE DIGESTION RUNS

Mesophilic acid-phase runs						
Run no.	AP2M7	AP2M6	AP2M5.5	AP2M5	AP1.3M7	AP1.3M5
Digester no.	334	334	334	334	334	334
Culture temperature, °C	35	35	35	35	35	35
Culture pH	7	6	5.5	5	7	5
HRT, days	2	2	2	2	1.3	1.3
Organic loading rate, kg VS/m^3-day	25.0	25.0	25.0	25.0	38.5	38.5
Feed volatile solids concentration, g/L	50.0	50.0	50.0	50.0	50.0	50.0
Thermophilic acid-phase runs						
Run no.	AP217	AP2T6	AP2T5.5	AP2T5	AP1.3T7	AP1.3T5
Digester no.	335	335	335	335	335	335
Culture temperature, °C	55	55	55	55	55	55
Culture, pH	7.0	6.0	5.5	5.0	7.0	5.0
Culture temperature, °C	55	55	55	55	55	55
HRT, days	2.0	2.0	2.0	2.0	1.3	1.3
Organic loading rate, kg VS/m^3-day	25.0	25.0	25.0	25.0	38.5	38.5
Feed VS concentration, g/L	50.0	50.0	50.0	50.0	50.0	50.0

TABLE 9. STEADY-STATE OPERATING CONDITIONS FOR ADVANCED STUDIES FOR MESOPHILIC (BOTH PHASES) TWO-PHASE DIGESTION RUNS

Run no.	UTP7M-M	TP3M-M(E)
Digester no.(s)	338-339	334-333
Culture mode	Upflow	CFCSTR
Feed pretreatment	None	Enzymatic
HRT, days		
Acid-phase	2	0.8
Methane-phase	5	2.2
System	7	3.0
Loading rate, kg VS/m^3-day	7.1	16.7
Feed volatile solids concentration, g/L	50.0	50.0

SECTION 6

MATERIALS AND METHODS

PROCESS FEEDS

Mixed primary-activated municipal sludges were used as digester feeds in this project. These mixed sludges were either obtained directly from a treatment plant or were prepared by mixing activated and primary sludges collected separately. Raw sludges were obtained from several sources and were processed by various methods, as described in the following sections, to prepare feedstocks for freezer storage. Digester feed slurries were prepared from these homogenized batches of feedstocks.

Sources of Raw Sludge

Raw wastewater sludges were collected from several water pollution control plants in the Greater Chicago area to conduct the digestion experiments. Raw primary, raw activated, and raw mixed primary-activated sludges were collected from wastewater treatment plants located in Hanover Park, Chicago, and Downers Grove, Illinois.

The fundamental studies digestion runs were conducted with mixed primary-activated sludges collected from the Hanover Park wastewater treatment plant of the Metropolitan Sanitary District of Greater Chicago (MSDGC). The Hanover Park plant is located in a northwest suburb of Chicago, and has a wastewater flow rate of about 35,000 m^3/day (9.2 mgd). About 5% of the plant flow is from industrial discharges. The collected sludge contained about 60% primary sludge and 40% activated sludge.

The applied studies were conducted with raw sludges from wastewater treatment plants in Hanover Park and Downers Grove, and with vacuum-filtered activated sludge cake from the West-Southwest (Stickney) wastewater treatment plant of the MSDGC. The Stickney plant has a raw sewage flow of about 820 MGD; about 50% of this flow is of industrial origin. Vacuum-filtered activated sludge cakes having TS contents between 12 and 14 wt % were collected. The Downers Grove plant has a wastewater flow rate of about 42,000 m^3/day, and 10%-15% of the flow is of industrial origin. Raw primary sludge having a solids content between 3.5 and 5 wt % was collected from this plant.

Collection and Processing of Hanover Park Raw Sludge

Raw municipal sludge that could be collected from the Hanover Park wastewater treatment plant was dilute in solids content and was not suitable for use as digester feed directly. This sludge had to be concentrated before digester feed could be prepared. Four different processing methods, described below, were used to concentrate the Hanover Park sludge.

Method 1. Freezing and Thawing in 200-Liter Drums--

The raw sludge was collected in large lots of several 200-liter drums from the Hanover Park plant. The collected sludge was trucked immediately to a freezer warehouse and stored at -26°C. Several drums of the frozen sludge from a lot were retrieved at a time from the cold storage and brought to IGT for thawing and solid-liquid separation upon completion of the thawing process. The liquid portion (supernatant) was decanted and discarded, and the concentrated bottom sludges from all the drums were fed to a 0.6-m^3-capacity double-ribbon blender and homogenized to prepare a single batch. While blending was in progress, sludge was withdrawn from a bottom port in the blender to fill up 10-liter plastic storage bags. Small aliquots taken from each bag were mixed in a container to produce a composite sample for total solids (TS), volatile solids (VS), and other analyses. The 10-liter sludge containers were stored in the refrigerated warehouse or in an IGT freezer; these containers were thawed, as needed, and the concentrated sludge was diluted and blended with tap water to prepare digester feed sludge of a selected consistency. The feed slurry was stored in a refrigerator. It was charged directly to the digester for manual (and once-a-day) feeding. For continuous feeding, the feed sludge was stored in a refrigerated in a continuously mixed feed reservoir, and this sludge was delivered to the digester with a timer-operated pump.

Method 2. Freezing and Thawing in 10-Liter Bags--

Method 2 was used in a few cases when digester feed sludge was needed quickly. In this method, the collected lot of raw dilute sludge was brought to IGT, and several drums were blended in the 0.6-m^3-capacity blender. The blended sludge was transferred to 10-liter plastic bags, which were then placed in an in-house freezer. The bags were then thawed; water was drained out of the bag during the thawing, leaving concentrated sludge in the bag. The contents of the bag(s) were homogenized to produce a batch of thick sludge.

Method 3. Concentration by Laboratory Centrifuge--

In Method 3, a laboratory centrifuge (Sorvall® Model RC-5B) was used to concentrate small lots of raw sludge. Centrifugation was done for 10 minutes at 7000 rpm, and the centrifuged pellets were blended to prepare a concentrated batch of sludge. Since only 3 liters of sludge could be processed at a time by the laboratory centrifuge, this method was used when feed sludge was needed quickly.

Method 4. Concentration by Pilot Centrifuge--

Considering the tedious and time-consuming nature of sludge concentration by freezing and thawing, an alternative sludge concentration procedure involving the use of a pilot centrifuge was tried during the second year of the project. The dilute raw sludge was concentrated by a Model 309 Alfa-Laval pilot centrifuge with a rated capacity of 4-40 liters/min. The centrifuge was fed continuously with an air-operated drum pump at flow rates considerably below 40 liters/min. Many problems, including centrifuge-motor burn out, pipe clogging, etc., were experienced. In addition, the solids capture in the centrifuged cakes was low. It was felt that considerable amounts of biodegradable solids were probably lost in the discarded supernatant. The solids content of the centrifuged cake was about 8.5 wt % TS without any polymer treatment of the dilute sludge. Only one lot of sludge was processed by this method.

Nineteen lots of Hanover Park raw sludge, totaling about 30,000 liters were collected, during the project. The sludge lots were processed in 1 to 15 batches. The processing method for each batch is described in the next section. The raw sludge had TS contents between 2 and 3.5 wt % and VS contents between 68 and 76 wt % of TS. By comparison, the concentrated sludge had VS concentrations between 60 and 79 wt % of TS.

Collection and Processing of Stickney and Downers Grove Sludges

Since the processing of the mixed activated-primary sludge from Hanover Park was difficult, digester feed sludge was prepared by mixing concentrated activated and primary sludges. Activated sludge (AS) cakes were collected from the wastewater treatment plant of Chicago Metropolitan Sanitary District in Stickney, IL. These sludge cakes had a TS concentration of about 14 wt % (Table 10), so there was no need for sludge concentration. The collected sludge cakes were diluted and blended in a commercial-size double-ribbon blender at IGT, and the blended sludges were transferred to 10-liter plastic bags for storage at -26°C.

TABLE 10. COLLECTION, PROCESSING, AND SOLIDS ANALYSES OF VACUUM-FILTERED ACTIVATED SLUDGE CAKE FROM STICKNEY

Date collected	Lot no.	Quantity, kg	TS, wt %	VS, wt % of TS
9/84	20	221	13.67	65.68
11/84	21	314	13.67	66.55

Raw primary sludges (PS) were collected from the Downers Grove wastewater plant; these sludges had TS contents between 3.5 and 5 wt % (Table 11). The VS concentration of the Stickney activated sludge was between 65% and 67% of TS. By comparison, the VS concentration of the Downers Grove sludge varied between 75% and 80% of TS. The activated and primary sludges were mixed in the ratio of about 73% activated to 27% primary sludge on a dry TS basis — similar to the AS/PS ratio of 75:25 for the Hanover Park sludge — to provide

TABLE 11. COLLECTION, PROCESSING, AND SOLIDS ANALYSES OF DOWNERS GROVE PRIMARY SLUDGE

Date collected	Lot no.	Quantity, liters	TS, wt %	VS, wt % of TS
10/84	22	49	4.90	79.67
12/84	23	58	3.78	75.86
12/84	24	15	4.63	77.82
12/84	25	18	4.34	76.71

about a 7 wt % TS-content digester feed sludge. The digester feed sludge was prepared in this manner to provide a VS concentration similar to that of the Hanover Park sludge (68% to 70% of TS).

APPARATUS FOR DIGESTION SYSTEMS

Eight digestion systems were used to conduct the Fundamental and Advanced Studies digestion runs. The digester dimensions and volumes are described in Tables 12 through 15. Details of the digester systems and ancillary equipment are described below.

Digestion Systems for Fundamental Studies

Six digesters were used to conduct the CFCSTR single-stage and two-phase process-comparison runs (Tables 12 and 13) and the CFCSTR acid-phase parametric-effects runs (Table 14). These runs were part of the fundamental studies. All the digesters were cylindrical in shape and fabricated of Plexiglas with removable headplates. Various ports were provided for feed slurry delivery, effluent removal, and sampling of the liquid and gaseous digester effluents. Each digester was provided with equipment for culture agitation, temperature control, and gas collection. Automatic feed systems were installed on the single-stage and acid-phase digesters to permit intermittent feeding under conditions approximating continuous flow. In addition, automatic pH controllers were installed on the two separate acid-phase digesters to control culture pH's. A schematic diagram of a CFCSTR two-phase digestion system is presented in Figure 9; the design of the single-stage and parametric-effect acid-phase digesters was similar to that shown in Figure 9 except that these systems had no methane-phase digester.

Each digester was mixed with three-blade propellors mounted on a stainless-steel shaft, which passed through a custom-made shaft-seal housing mounted on the center of the digester headplate. The shaft was driven at

TABLE 12. LIST OF CFCSTR DIGESTERS USED FOR SINGLE-STAGE DIGESTION RUNS FOR PROCESS COMPARISON STUDIES

Run no.	Digester no.	Digester height, cm	Digester diameter, cm	Total volume, L	Culture volume, L
Mesophilic (35°C)					
SS15M	331	38	29	25.5	20.0
SS7M	331	38	29	25.5	15.0
SS3M	331	38	29	25.5	15.0
Thermophilic (55°C)					
SS15T	337	38	29	25.5	20.0
SS7T	331	38	29	25.5	15.0
SS3T	335	25	19	7.2	5.2

about 120 rpm by a variable speed motor. Four vertical baffles were attached 90° apart on the inside wall of the digester to minimize vortexing.

Digester temperature was sensed by a thermistor probe installed in an oil-filled thermowell, which extended through the digester headplate into the culture. The temperature was maintained by a proportional controller connected to heating tapes or pads wrapped around the outside of the digester. The controller maintained the culture temperature to within 0.5°C of the setpoint.

Gas production in each digester was measured with automatic gas burets, which continuously collected and wasted small volumes of gas (about 50 mL) as it was produced by the digester. Gas collection and wasting cycles were totalized by electro-mechanical digital counters. Gas productions were determined to an accuracy of 3% or less, as determined by periodic calibrations.

Each automated feed system consisted of a refrigerated and mixed 19-liter feed reservoir, a timer-operated progressive cavity pump and associated piping and valves (Figure 9). The feed reservoir was maintained at 3° to 5°C to minimize biological degradation of the feed slurry. A pneumatically operated pinch valve was installed between the pump and the digester to prevent backflow of the culture through the pump. The autofeed system delivered feed slurry to the digester at regular intervals (12 to 60 times per day) to approximate continuous-flow conditions.

TABLE 13. LIST OF CFCSTR DIGESTERS USED FOR TWO-PHASE DIGESTION RUNS FOR PROCESS COMPARISON STUDIES

Run no.		Digester no.	Digester height, cm	Digester diameter, cm	Total volume, L	Culture volume, L
Meso-meso						
TP15M-M	Acid-phase	332	25	19	7.2	3.0
	Methane-phase	333	38	29	25.5	20.0
	System	--	--	--	32.7	23.0
TP7M-M	Acid-phase	334	25	19	7.2	5.2
	Methane-phase	333	38	29	25.5	13.5
	System	--	--	--	32.7	18.7
TP3M-M	Acid-phase	334	25	19	7.2	5.2
	Methane-phase	333	38	29	25.5	12.3
	System	--	--	--	32.7	17.5
Meso-thermo						
TP15M-T	Acid-phase	334	25	19	7.2	3.2
	Methane-phase	337	38	29	25.5	20.0
	System	--	--	--	32.7	23.2
TP7M-T	Acid-phase	334	25	19	7.2	5.2
	Methane-phase	331	38	29	25.5	15.0
	System	--	--	--	32.7	20.2
Thermo-thermo						
TP7T-T	Acid-phase	335	25	19	7.2	5.2
	Methane-phase	331	38	29	25.5	15.0
	System	--	--	--	32.7	20.2
TP3T-T	Acid-phase	335	25	19	7.2	5.2
	Methane-phase	331	38	29	25.5	15.0
	System	--	--	--	32.7	20.2

TABLE 14. LIST OF DIGESTERS USED FOR CFCSTR PARAMETRIC-EFFECTS ACID-PHASE DIGESTION RUNS

Run no.	Digester no.	Digester height, cm	Digester diameter, cm	Total volume, L	Culture volume, L
Mesophilic					
AP2M7	334	25	19	7.2	6.0
AP2M6	334	25	19	7.2	5.2
AP2M5.5	334	25	19	7.2	5.2
AP2M5	334	25	19	7.2	5.2
AP1.3M7	334	25	19	7.2	5.2
AP1.3M5	334	25	19	7.2	5.2
Thermophilic					
AP2T7	335	25	19	7.2	6.0
AP2T6	335	25	19	7.2	5.2
AP2T5.5	335	25	19	7.2	5.2
AP2T5	335	25	19	7.2	5.2
AP1.3T7	335	25	19	7.2	5.2
AP1.3T5	335	25	19	7.2	5.2

Effluents from the automatically-fed single-stage and parametric-effects acid-phase digesters were wasted continuously through gravity overflow pipes to collection vessels. The overflows were U-shaped in design to prevent the loss of product gases with the effluent. Effluents from the acid-phase digester of the two-phase system were delivered to their associated methane-phase digester by gravity overflows or by timer-operated peristaltic pumps.

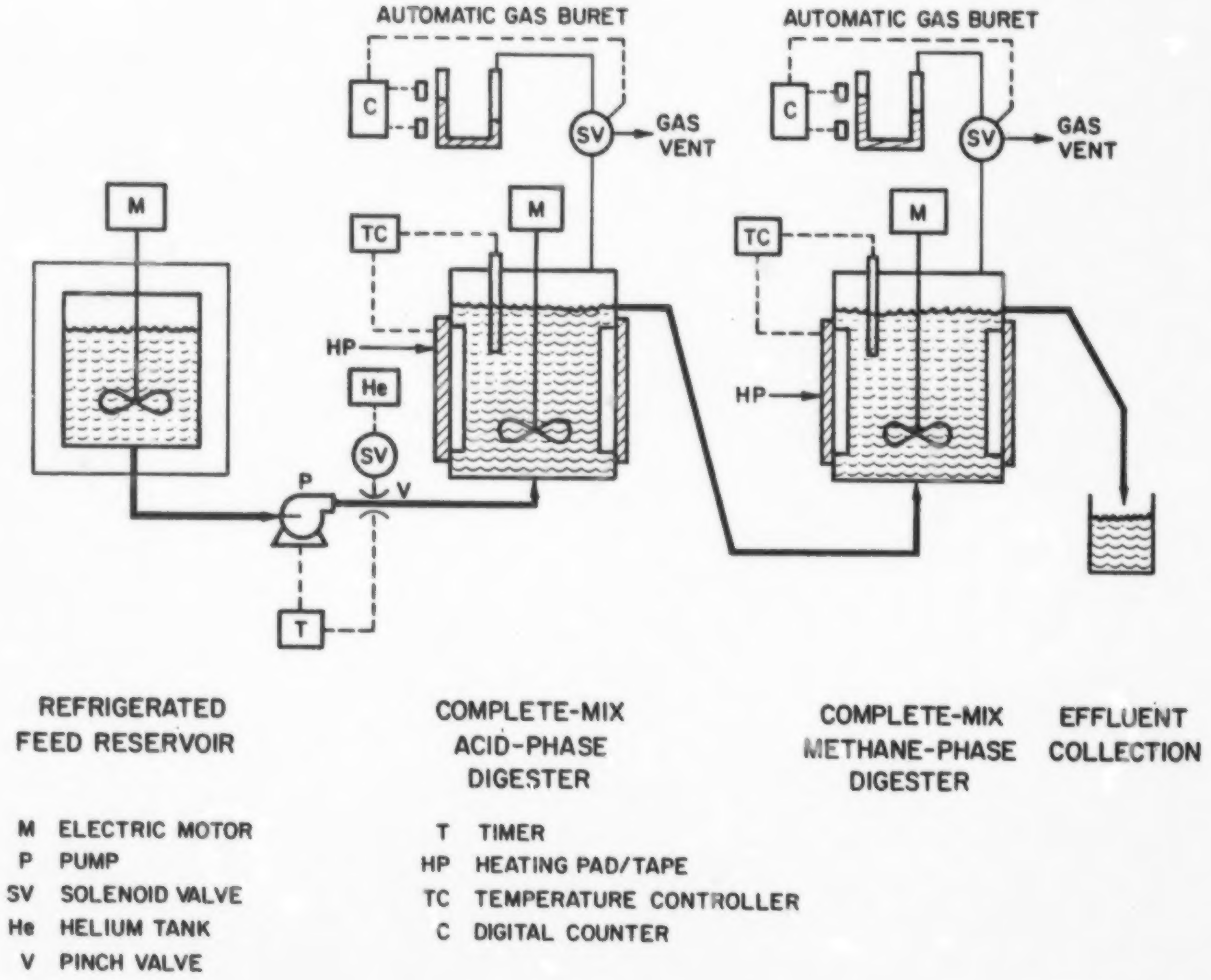
The pH control systems for the parametric-effects acid-phase digesters consisted of an automatic pH controller with analog pH meter (Cole-Parmer Model No. 5997), an in-line pH probe, and a peristaltic pump and storage vessel for delivery of the pH control solution to the culture. The pH probe was mounted through the headplate and extended about 2.5 cm deep into the culture. The peristaltic pump was activated by the controller to deliver the pH control solution to the digester whenever the culture pH deviated more than about 0.1 pH units from the setpoint. A 2.5-N solution of NaOH was used to control the culture to pH 7; 2.5-N HCl was used for control to pH 6 and below.

TABLE 15. LIST OF DIGESTERS USED FOR ADVANCED TWO-PHASE DIGESTION RUNS

Run no.		Digester no.	Culture mode	Digester height, cm	Digester diameter, cm	Total volume, L	Culture volume, L
CPCSTR two-phase system operated with enzyme-treated sludge							
TP3M-M(E)	Acid-phase	334	CFCSTR	25	19	7.2	5.2
	Methane-phase	333	CFCSTR	38	29	25.5	12.9
	System	—	--	--	--	32.7	16.1
Upflow two-phase system							
UTP7M-M	Acid-phase*	338	Upflow	--	--	8.5	7.0
	Methane-Phase†	339	Hybrid upflow/ complete-mix	71	19	21.5	19.0
	System	—	--	--	--	30.0	26.0

* The acid-phase digester was rectangular in shape with a trapezoidal bottom with a height, width, and length of 22, 11, and 34 cm, respectively.

† The methane-phase digester was cylindrical in shape with a small inverted pyramid at the bottom, 20 cm square and 9 cm high.


AUTOMATIC GAS BURET
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C
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V
T
REFRIGERATED FEED RESERVOIR
COMPLETE-MIX ACID-PHASE DIGESTER
COMPLETE-MIX METHANE-PHASE DIGESTER
EFFLUENT COLLECTION
M ELECTRIC MOTOR
P PUMP
SV SOLENOID VALVE
He HELIUM TANK
V PINCH VALVE
T TIMER
HP HEATING PAD/TAPE
TC TEMPERATURE CONTROLLER
C DIGITAL COUNTER

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Figure 9. Schematic diagram of CFCSTR two-phase anaerobic digestion system.

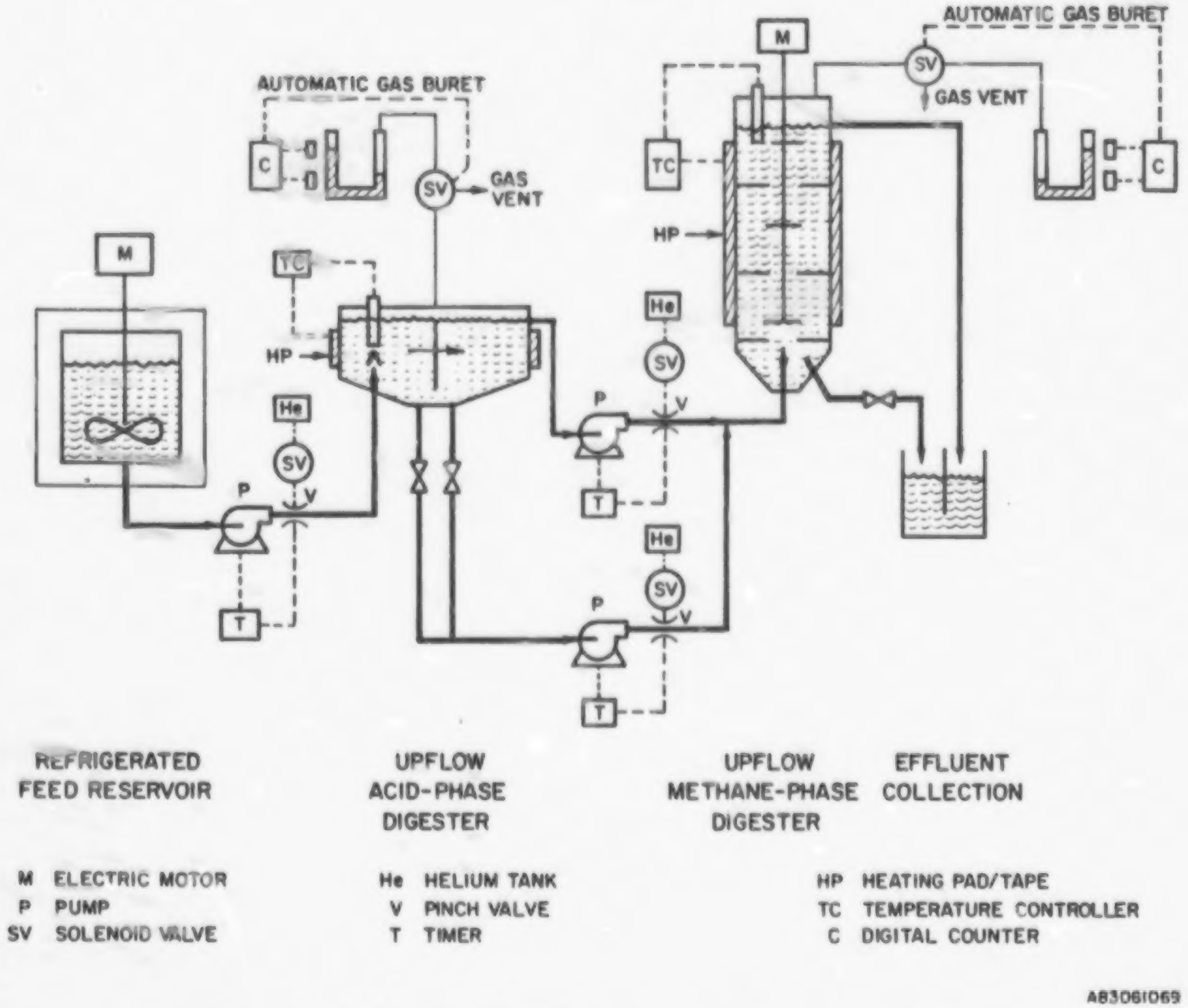
Digestion Systems for Advanced Studies

Four digesters comprising two separate two-phase systems were used to conduct the advanced studies digestion runs (Table 15). One of the two-phase systems (Digester Nos. 333 and 334) was operated as a CFCSTR system receiving enzyme-treated sludge, and the other (Digester Nos. 338 and 339) was operated as an upflow system. Systems for culture agitation, temperature control, gas collection, feed slurry delivery, and effluent removal were similar to those described previously for the fundamental studies runs, except as noted below.

A lipase dosing system was installed on the acid-phase digester (Digester No. 334) of the two-phase system operated with enzyme-treated sludge feed. The dosing system consisted of a timer-operated peristaltic pump and an enzyme storage vessel. The rate and frequency of delivery of the lipase solution to the acid-phase digester was controlled by varying the settings of the timer.

A schematic of the upflow two-phase system is depicted in Figure 10. The upflow acid-phase digester (Digester No. 338) consisted of a rectangular tank separated into two compartments by a central vertical baffle. The digester feed slurry was pumped into the left compartment through a vertical feed pipe that extended through the bottom of the digester about halfway up into the culture. A small deflector located just above the feed pipe directed the slurry toward the bottom of the compartment. Feed solids then flowed upward toward the culture surface in the left compartment, over the central baffle, and into the right compartment. Additional baffles (not pictured) were installed in the right compartment to keep floating scum away from the overflow effluent port and to promote sedimentation and retention of feed solids within the digester. The bottom of each compartment sloped toward the center of the digester to permit storage of settled solids. Two pipes with ball valves were installed at the bottom of the digester on either side of the central baffle and were connected to a pump to permit delivery of the stored sludge to the methane-phase digester. Sludge could be withdrawn from either compartment by opening one of the ball valves and closing the other. The rate of sludge withdrawal from the bottom of the acid-phase digester was controlled by a timer that operated the pump. Temperature control was provided by hot water, which was recirculated from a water bath through a jacket around the outside of the digester.

The upflow methane-phase digester (Digester No. 339) consisted of a cylindrical tank with a truncated, inverted pyramid-shaped bottom, a feed pipe and solids deflector, and three sets of alternating static baffles and turbine-type impellors. Effluents from the acid-phase digester were pumped into the bottom of the methane-phase digester through a vertical pipe which extended about one-third up into the culture. The solids in the incoming feed were directed toward the bottom of the digester by an inverted cup-shaped deflector that allowed the liquid portion of the feed to flow upward toward the culture surface. Three ring-shaped horizontal baffles were attached at the 9, 13, and 17-liter levels of the digester. In addition, three sets of turbine-type impellors were attached to a low-speed (9.8 rpm) central rotating shaft. This pattern of alternating static baffles and impellors was designed to produce a circuitous flow pattern within the digester while also providing areas of local mixing. Effluent ports with U-shaped overflows were installed


AUTOMATIC GAS BURET
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REFRIGERATED FEED RESERVOIR
UPFLOW ACID-PHASE DIGESTER
UPFLOW METHANE-PHASE DIGESTER
EFFLUENT COLLECTION
M ELECTRIC MOTOR
P PUMP
SV SOLENOID VALVE
He HELIUM TANK
V PINCH VALVE
T TIMER
HP HEATING PAD/TAPE
TC TEMPERATURE CONTROLLER
C DIGITAL COUNTER
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Figure 10. Schematic diagram of two-phase upflow digestion system for applied studies.

at the side of the digester to permit operation at culture volumes of 15 to 19 liters.

CHEMICAL ANALYSES

Sample Collection and Preparation

Sample Collection--

Sample collection was carried out in strict accordance with the sample collection procedures outlined in Part 105, APHA Standard Methods (15th Edition)[58] or in ASTM Part 26, (1982) manuals.[59] Two types of samples, gas and liquid, were collected. Gas samples were collected directly from the digester head space in well-purged disposable plastic syringes and analyzed immediately by gas chromatography. Liquid samples were collected during sludge processing operations and from digester feed reservoirs and effluents.

As specified in Table 16, three types of liquid samples were collected: grab, grab composite, and time composite.

Grab Samples--Grab samples were spot or catch samples representing physical and/or chemical characteristics at the time of sampling and the location of sample collection.

Grab Composite Samples: Processed Feed--

Several constant-volume grab samples were collected from each large batch of blended feed sludge, which was withdrawn from a large blender to fill 10 or 20-liter containers for refrigerated storage. The grab samples were collected every time a sludge container was filled. These samples were mixed and homogenized to produce a grab composite for solids analyses.

Grab Composite Samples: Feed and Effluent Slurries--Daily grab samples from digester feed reservoirs and effluent were collected, composited, and processed to characterize feed and effluent quality during steady-state segments of the digestion runs.

Time Composite Samples: Effluents--Grab samples were composited from digester effluents accumulated over a 24-hour period.

Sample Preparation--

Sample preparation was not required for gas samples; these were analyzed immediately after collection. The liquid samples were homogenized for representativeness of the final aliquots withdrawn for analysis. Wherever possible, sample preparation procedures as outlined in the APHA or ASTM Standard Methods were followed.

In the case of the COD and ammonia and organic nitrogen analyses, sample preparation procedures were modified to improve the accuracy and precision of these analytical determinations. These modifications were necessary due to the heterogeneous nature and high solids contents of the slurries.

TABLE 16. SAMPLE COLLECTION AND PROCESSING PROTOCOL

Determination	Sample source	Sample type	Sample preservation (if not analyzed immediately)
Carbon (total)	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Hydrogen	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Nitrogen, ammonia	Influent (feed)	Grab, grab composite	pH <2 with H_2SO_4, seal and freeze
	Effluent	Grab, grab composite, time composite	pH <2 with H_2SO_4, seal and freeze
Nitrogen, organic	Influent (feed)	Grab, grab composite	pH <2 with H_2SO_4, seal and freeze
	Effluent	Grab, grab composite, time composite	pH <2 with H_2SO_4, seal and freeze
Sulfur (total)	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Phosphorus	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Ash	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Heating value	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Total solids	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Volatile solids	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Fixed solids	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
pH	Influent (feed)	Grab	--
	Effluent	Grab	--
Alkalinities (total and bicarbonate)	Influent (feed)	Grab	--
	Effluent	Grab	--
Volatile acids	Influent (feed)	Grab	1.5 mL 20% H_3PO_4/10 ml sample; refrigerate at 4°C
	Effluent	Grab	1.5 mL 20% H_3PO_4/10 ml sample; refrigerate at 4°C
COD (total and filtrate)	Influent (feed)	Grab, grab composite	pH <2 with H_2SO_4, seal and freeze
	Effluent	Grab, grab composite, time composite	pH <2 with H_2SO_4, seal and freeze
Lipids	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Carbohydrates (total)	Influent (feed)	Grab, grab composite	Seal and freeze
	Effluent	Grab, grab composite, time composite	Seal and freeze
Gas composition	Head space	Grab	--

Total COD analyses were conducted with feed and effluent slurries from two of the CFCSTR parametric-effect acid-phase runs using APHA Standard Methods and modified sample preparation procedures to compare the precision of the two methods (Table 17). Analyses of the samples were conducted in an identical manner with regard to reagent volumes and reflux time and differed only in the preparation method.

Preparation and dilution of samples by the APHA method (Method 1) consisted of the following steps:

- The collected sample (about 500 mL) was homogenized for about 1 minute in a Waring blender.
- 5.0 mL of blended and mixed sample were transferred to a volumetric flask with a 5.0-mL wide-tip pipet and diluted to 500.0 mL with distilled water to prepare a 1:100 diluted sample.
- 10-mL aliquots of the mixed and diluted sample were added to each COD flask with a 10.0 mL wide-tip pipet.

Sample preparation and dilution by the modified method (Method 2) consisted of the following steps:

- The collected sample was homogenized as in Method 1.
- About 25 g of blended and mixed sample was weighed and diluted with 25.0 mL distilled water to prepare a dilution of known proportion.
- Four 5-mL aliquots of this diluted and mixed sample were diluted to 500.0 mL in a 500.0 mL volumetric flask; the pipets were flushed into the flask with distilled water after the transfer of each aliquot.
- Two 5.0-mL aliquots of this diluted and mixed sample were pipeted into each replicate COD flask; each aliquot was flushed with distilled water into the COD flask after transfer.

The standard deviations of the COD's conducted by the modified sample preparation method were substantially lower than those conducted by the APHA sample preparation method, for both feed and effluent slurries. The data indicated that the precision of total COD's on samples prepared by the modified method was superior to that obtained with samples prepared by the APHA method. Accordingly, all of the total COD analyses were conducted with samples prepared by the modified method. Samples analyzed for ammonia and organic nitrogen were also prepared by this method.

Sample Handling, Identification, Preservation, and Storage

Liquid samples were collected in clean glass and plastic bottles that were rinsed out two or three times with the fluid being sampled. Before collecting samples from tubings or pipes, the lines were flushed out sufficiently by draining the digester or reservoir contents to ensure collection of representative samples. Each sample bottle was marked with an

TABLE 17. EFFECT OF SAMPLE PREPARATION ON TOTAL COD DETERMINATIONS OF FEED AND EFFLUENT SLURRIES FROM CFSTR MESOPHILIC AND THERMOPHILIC ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE

	Run AP2T7				Run AP2M7			
	Feed slurry		Effluent slurry		Feed slurry		Effluent slurry	
Sample preparation method	APHA*	Modified†	APHA	Modified	APHA	Modified	APHA	Modified
Total COD, mg/L								
Replicate 1	103,054	77,655	101,322	68,842	69,820	81,972	99,590	88,268
Replicate 2	127,302	78,994	99,590	69,706	107,384	86,164	113,446	82,298
Replicate 3	139,426	80,780	51,094	71,152	97,858	86,630	64,950	82,298
Replicate 4	--	79,887	--	74,387	--	95,945	--	84,430
Replicate 5	--	80,333	--	76,235	--	84,767	--	--
Average	123,260	79,530	84,002	72,064	91,507	87,096	92,662	84,323
Standard deviation	18,520	1,239	28,512	3,145	19,830	5,269	24,979	2,815
Coefficient of variation, %	15.0	1.6	33.9	4.4	21.7	6.0	27.0	3.3

* The APHA sample preparation consisted of preparing a 1:100 dilution of the sample; 10.0-mL aliquots of the diluted and mixed sample were added to each replicate flask with a 10-mL wide-tip pipet.

† The modified sample preparation consisted of diluting a known weight of sample (about 25 grams) with 25.0-mL of water; two 5.0-mL aliquots of this diluted and mixed sample were added to a 500.0-mL volumetric flask with a 5.0-mL wide-tip pipet, and two 5.0 ml aliquots of this mixed and diluted sample were added to each replicate flask. Each aliquot was washed with water directly into the flask after pipeting.

identification number; a label containing information on date, time, location of sample, and the name of the sample collector was securely attached.

All samples were analyzed for the intended data as soon as they were collected, if possible. Temperature, pH, alkalinity, and gas composition determinations were performed immediately after collection of the sample. In cases where the analysis could not be performed immediately, the sample was preserved by procedures prescribed in APHA (15th Edition)[58] and ASTM (1982) Standard Methods.[59]

Feed Analyses

Processed sludge feed lots were analyzed for total solids (TS), volatile solids (VS), and fixed solids (FS). Digester feed slurry samples collected during steady state were analyzed for pH, TS, VS, and FS, total and bicarbonate alkalinities, ammonia and organic nitrogen, lipids, carbohydrates, and crude protein. Several fresh slurry samples were also analyzed for total and volatile suspended solids and total and filtrate COD, elemental content, and heating value. An anaerobic biogasification potential (ABP) test was also conducted on one Hanover Park feed sample to estimate the biodegradability of the feed sludge.

Effluent Analyses

Digester performances were monitored by daily measurement of gas production; effluent pH was determined at least three times per week. Gas composition and volatile acids concentrations were analyzed at least once per week during nonsteady-state segments and two or three times per week during steady-state segments. Steady-state effluent samples were analyzed for TS, VS, and FS, total and bicarbonate alkalinities, ammonia and organic nitrogen, lipids, carbohydrates, and crude protein. In a few cases, effluents were also analyzed for total and volumetric suspended solids, total and filtrate COD, elemental contents, and heating value.

Analytical Procedures

A number of physical and chemical analyses were performed to evaluate digester performance, monitor the progress of digestion runs, and characterize digester feeds and effluents. Details of these analytical methods are presented in Table 18.

Information derived from results of the physical and chemical analyses was used for the following purposes:

- To determine the empirical formula of the feed, and to estimate theoretical and biodegradable gas and methane yield potentials
- To monitor acid- and methane-phase culture development, population transitions in response to changes in operating conditions, and the condition of the culture during runs

TABLE 18. LIST OF PHYSICAL AND CHEMICAL ANALYSES/MEASUREMENTS, METHOD OF DETERMINATION, AND THE INTENDED USE OF THE RESULTING DATA

Measurement/Analysis	Sample	Method	Intended Use
Temperature	Digester culture	APHA Std. Methods (15th ed.) Part 212	Culture monitoring and maintenance; evaluate temperature effect on digestion.
	Digester gas	Same as above	Reduce gas volumes to those at the standard temperature of 60°F.
Liquid flow rate	Digester influent and effluent	Independent determination of liquid volume in calibrated containers and time by certified timers	Determine residence times; use to perform mass balances.
Gas volume	Digester gas	Measured in a gas collector calibrated according to ASTM Part 26, Designation D-1071-780, Article 12 (1982)	Calculate total gas yield and methane yield and determine process efficiency.
Gas pressure	Digester gas	ASTM 1982 edition Part 26 (1982) Designation D-3631-77	Reduce gas volumes to those at the standard pressure of 30-in. Hg.
C, H, N, S, Ash, Heating Value, and P	Digester feed and effluent	ASTM 1982 edition, Part 26, Designation D-3178, 3179, 3177, 3174, and APHA Std. Methods (15th ed.) Part 424	Estimate empirical formulas and theoretical gas and methane yields; elemental balances; use in estimating carbon and energy recoveries in gas; nutrient availability and limitations.
TS, VS, Fixed solids	Same as above	APHA Std. Methods (15th ed.) Part 209-G	Determine gas and methane yields; solids balances; estimate sludge concentration and SRT; determine solids reduction efficiencies.
Total and volatile suspended solids	Digester feed and effluent	Centrifugation method (described in text)	Determine suspended solids retention and liquifaction.
pH	Same as above	APHA Std. Methods (15th ed.) Part 423	Culture monitoring and maintenance; evaluate pH effect.
Total and bicarbonate alkalinity	Same as above	Total alkalinity as per APHA Std. Methods (15th ed.) Part 403-4; bicarbonate alkalinity by calculation	Culture monitoring, maintenance, and status; use in carbon balance.
Organic-N	Same as above	APHA Std. Methods (15th ed.) Part 420-A	Estimate crude protein and protein digestion efficiency.
Ammonia-N	Same as above	APHA Std. Methods (15th ed.) Part 417-D	Monitor nitrogen availability; use in nitrogen balance.
Volatile Acids	Digester feed and effluent	Modified APHA Std. Methods (15th ed.); procedure modification described in text	Estimate feed-to-acid and acid-to-gas conversion efficiencies.
	Digester culture	Same as above	Monitor digestion status.
COD (total and filtrate)	Digester feed and effluent	APHA Std. Methods (15th ed.) Part 508-A	Evaluate feed hydrolysis and acidification efficiencies; estimate acids-to-methane conversion efficiency.
Lipids	Same as above	APHA Std. Methods (15th ed.) Part 503-D	Evaluate lipid conversion efficiency.
Crude protein	Same as above	Organic nitrogen X 6.25	Evaluate protein digestion efficiency.
Carbohydrate	Same as above	American Society for Microbiology, Manual of Methods for General Microbiology (1981)	Evaluate carbohydrate conversion efficiency.
Gas production	Digester gas	Same as gas volume	Determine gas and methane yields and production rates; utilize to conduct mass balance.
Gas composition (CH_4, CO_2, H_2, N_2)	Same as above	APHA Standard Methods (15th ed.) Part 511-B	Monitor culture status; determine methane yields; utilize in mass balances

- To identify steady-state operation and evaluate steady-state digester performances and efficiencies in accordance with project objectives

- To evaluate conversion efficiencies of various feed components, and to perform mass balances

- To determine substrate conversion and product formation efficiencies during steady-state segments of the digestion runs.

Standard Test Procedures

As indicated in Table 18, measurement methods and test procedures used throughout this project were as specified in APHA Standard Methods, ASTM, or other accepted analytical manuals, with the exceptions of the volatile fatty acids (VFA) and suspended solids determination. Descriptions of these modified procedures are provided below.

Evaluation and Selection of Alternative Analytical Procedures

Volatile Fatty Acids—

Gas chromatography was used to determine VFA concentrations in feed and digested sludges. The chromatographic technique was preferred to the elution chromatographic and distillation methods because, using this technique, the individual volatile acids could be separated and measured with greater accuracy and precision. Information on the concentrations of individual volatile acids was necessary for proper monitoring of digester performance; the gas chromatographic technique described here provided this required information. Also, the gas chromatographic method was less time-consuming and was convenient when a large number of samples had to be analyzed quickly. Most investigators in the anaerobic fermentation field utilize gas chromatographic techniques similar to the one described here to obtain volatile fatty acids concentration data.[60-64]

A Hewlett Packard Model 5840A Reporting Gas Chromatograph equipped with a hydrogen-air flame ionization detector, a programmable digital processor to control the various aspects of GC analysis (for example, temperatures, detector operation, integration of peak areas, component identification, chromatogram plotting, retention times, run programming, etc.), and an automatic liquid sampler were used. The glass column, 1.8 m x 4 mm in diameter, was packed with acid-washed Chromosorb 101, as suggested by All Tech Associates and John Mansville Corp.[65] Similar column packings are used by other investigators.[60-62] The injection port was maintained at 200°C, while the column was operated at 190°C. Detector temperature was set at 250°C. The nitrogen carrier gas was supplied at a flow rate of about 25 mL/min. Hydrogen gas pressure and flow rate were maintained at 18 psig and 25 mL/min, while air pressure and flow rate were set at 28 psig and 240 mL/min, respectively.

A 10-mL sample of the feed or digester effluent was acidified to a pH of about 1.7 with 1.5 mL of 20% H_3PO_4; it was then centrifuged for 15 minutes at 15,000 rpm to separate a clear liquid fraction, which was transferred to a 2-mL clean glass vial. The glass vial was sealed with a Teflon-faced rubber septum and then placed in an automatic sampler tray. A 1-μL sample was withdrawn from the bottle and injected into the chromatographic column by the automated injection system.

The injector was programmed for automatic washing to the 10-μL syringe with portions of the sample four times before the sample was withdrawn for injection and analysis. This operation was followed by eight additional purgings to ensure expulsion of air. The column was periodically injected with acidified water samples between sample injections. A standard VFA solution containing a mixture of individual volatile acids of known concentration was chromatographed in the same manner as the unknown. Unknown fatty acids concentrations were calculated by comparing areas under chromatogram peaks obtained for individual VFA's in the unknown and standard samples. Individual VFA concentrations are expressed in terms of equivalent acetic acid concentration.

Suspended Solids--

Analysis of feed and effluent suspended solids by the APHA Standard Methods procedure (Parts 209-D and 209-G, 15th Edition)[58] was problematic due to the high concentration (20 to 40 g/L) of suspended solids in the samples. To avoid clogging the filters with solids particles, it was necessary to use very small sample volumes (less than 5 mL). The difficulty in accurately measuring the small sample volumes resulted in widely dispersed replicate data for each determination. A centrifugation procedure was then developed for the determination of total and volatile suspended solids to overcome the limitations of the APHA filtration method.

Suspended solids determinations by the centrifugation method were conducted in triplicate and consisted of the following steps:

- Known weights of sample (about 20 g) were transferred to 50-mL centrifuge tubes.
- The tubes were centrifuged for 20 minutes at 20,000 rpm to separate the sample into solids pellets and clean supernate fractions.
- The clean supernate was drained off, taking care to minimize loss of floating solids particles; distilled water was then added to each tube, and the pellets were resuspended with a small magnet.
- The centrifugation and suspension steps were repeated twice more.
- The suspended solids were transferred to crucibles and dried and ashed at 103° and 550°C, respectively, to determine total and volatile suspended solids of the plug.
- The supernate collected after each centrifuge operation was analyzed for total and volatile suspended solids contents per APHA Standard Methods, Parts 209-D and 209-G.

- Suspended solids concentrations for each sample were reported as the sums of the TSS and VSS contents of the solids pellet and supernate fractions.

Carbohydrates--

Since the APHA Standard Methods do not include a carbohydrate analysis procedure for sewage sludge, and because there is no consensus among researchers as to which method of the many published techniques is suitable for this material, considerable work was done to select a suitable analytical procedure to determine the "total carbohydrate" contents of feed and digested sludges. As discussed below, determination of total carbohydrates in sludge is difficult because of the complex composition of this generic material, the heterogeneity of sludges, and the limitation of the analytical methods in detecting various types of sugars.

Carbohydrates can be classified as monosaccharides, oligosaccharides, and high-molecular-weight polysaccharides such as starch, glycogen, cellulose, hemicellulose, pectins, xylans, mannans, etc. In addition, sewage sludge contains a variety of carbohydrates that have their origin in microbial cells; this is particularly true of activated sludge. Cells contain nucleic acids that, upon degradation, yield deoxyribose and ribose. Microbial cell walls contain other complex polysaccharides (for example, capsular polysaccharides, lipopolysaccharide) containing amino sugars, and other monosaccharides.

Most commonly used analytical methods for determination of "total carbohydrate" are derived or adapted from the Molisch test.[66] This method involves heating the sample with concentrated sulfuric acid and a "color developer" which is usually an aromatic amine or a phenol. The reactions include —

- Hydrolysis of polysaccharides to monosaccharides
- Dehydration and transformation of the monosaccharide to form furfural (in the case of pentose sugars) or hydroxy methyl-furfural (in the case of hexose sugars)
- Complexation of the hydrolysis products with the color developer to form a colored compound, the concentration of which is measured spectrophotometrically.

Samples such as sewage sludge and digested sludge contain particulate polysaccharides of various compositions, and the result of total carbohydrate analysis depends to a large extent on the details of the hydrolysis procedure. The degree of hydrolysis and the nature of the hydrolysis products depend on the type of acid used, the acid concentration, pH, hydrolysis time and temperature, and other factors. For example, complete hydrolysis of cellulose yields glucose, whereas partial hydrolysis yields the disaccharide, cellulobiose. Hydrolysis of hemicellulose, on the other hand, produces D-xylose and D-glucoronic acids. The reactivities of these different compounds with the color developer are quite different. Also, it has been pointed out that certain hydrolysis products such as amino sugars, trioses, tetroses, and other carbohydrates that do not form furfural or furfural derivatives hardly react with the coloring agent.[67] Separate specific assays are, therefore, required to ascertain the concentrations of carbohydrates that do not yield furfurals upon hydrolysis. It is apparent that no single analytical method or color-forming agent can accurately measure the various types of carbohydrates that are present in biological suspensions and sewage sludges.[68]

Among the myriad of color developers such as indole, orcinol, carbazole, cysteine, trypotophan, α-naphthol, anthrone and phenol used for colorimetric determination of total carbohydrates, the last two compounds have been particularly useful and are utilized in the so-called anthrone and phenol-sulfuric-acid methods.[68] These two methods were investigated to select the better method for this project.

Analysis of the Hanover Park sludge with the anthrone and phenol-sulfuric acid methods indicated that a much higher concentration is obtained by the latter procedure (Table 19). One reason for the lower carbohydrate analysis by the anthrone method is that the anthrone reagent exhibits weak reactions with pentoses and heptoses, for example, and much stronger reactions with hexoses.[68] The phenol reagent, on the other hand, reacts equally well with all sugars. It was determined from HPLC analysis that the concentration of five carbon sugars was much higher than that of the hexoses, which could cause the anthrone analysis to be considerably lower than that obtained by the phenol-sulfuric acid procedure. Also, the total carbohydrate contents obtained by the anthrone procedure were much lower than those reported in the literature.[69-71]

TABLE 19. TOTAL CARBOHYDRATE CONCENTRATIONS IN HANOVER PARK SLUDGE AS DETERMINED BY THE ANTHRONE AND THE PHENOL-SULFURIC ACID METHODS

Analytical methods	Total carbohydrate concentration mg/L	Average	wt % of TS	Average	wt % of VS	Average
Anthrone	8,000		11.1		15.9	
	8,100	8,050	11.3	11.2	16.1	16.0
Phenol sulfuric	13,900		19.4		27.7	
	14,400		20.0		28.7	
	13,700		19.1		27.3	
	14,440	14,100	20.0	19.6	28.7	28.1

Lipids--

Considerable work was done to select a suitable procedure to determine lipid contents of feed and digested sludges. Lipids are a diverse group of high-molecular-weight carbon-oxygen-hydrogen compounds that are insoluble in water but soluble in such organic solvents as benzene, ethers (diethyl ether, petroleum ether, etc.), chloroform, acetone, pentane, hexane, freon (dichlorodifluoromethane, CCl_2F_2), or mixtures thereof. Simple lipids include fats (glycerides and triglycerides, which are products of a combination of fatty acids and the trihydroxyalcohol, glycerol), waxes (esters of fatty acids and alcohol rather than glycerol), oils (low- to high-molecular-weight hydrocarbons ranging from gasoline to heavy oil to lubricating oils), esters of long-chain fatty acids (calcium or magnesium soaps), etc. Compound lipids have a more complex structure and include phospholipids (for example, lecithins and cephalins, frequently combined with proteins) glycolipids, and sulfolipids. These compound lipids are present in all microorganisms and may yield nitrogenous bases, phosphoric acids, etc., in addition to fatty acids and glycerol upon hydrolysis. Derived lipids consist of a heterogeneous group of compounds derived from, or chemically related to, other lipids. These substances include steroids (hormones, ergosterols, cholesterols, etc.), cartenoids, and polyisoprenoids and behave like lipids in that they are extractible by lipid solvents. The feed and the digested sludges were expected to contain all types of lipids of natural and synthetic origins, although the relative proportions of the various lipid types are expected to be altered due to anaerobic fermentation.

Since all lipid analytical methods rely on its extraction by selected solvents, and because a chosen solvent does not selectively dissolve a particular kind of lipid, the determined concentration represents a heterogeneous group rather than a specific chemical classification. Lipid analytical methods are simple in principle and involve solvent extraction of this hydrophobic compound from an aqueous suspension followed by drying to produce a moisture-free residue. The methods are necessarily empirical; errors are introduced because low-boiling fractions are lost and certain non-lipid substances may be extracted along with lipids. Chloroform, for example, dissolves certain carbohydrates to a limited extent. Similarly, elemental sulfur and certain organic dyes are extracted as "hexane-soluble lipids." Special precautions are necessary because some extractibles, especially unsaturated fats and fatty acids, oxidize readily. However, replicable and comparable results can be obtained by strict adherence and meticulous attention to all procedural details. Consequently, the extraction technique and the rate and time of extraction must be reproduced exactly for all determinations because of varying solubilities of different kinds of lipids in the selected solvent. Also, the length of time for drying and the drying temperature (which influences volatilization) as well as the cooling time — excessive cooling time may result in an increase in lipid weight, presumably due to oxidation of extractibles and the absorption of oxygen by the solvent — must be closely controlled and kept constant to produce comparable and meaningful results.

Because there appears to be no consensus as to which of the various lipid analytical procedures is "best" for sewage sludges, three methods were investigated. One of these methods was that recommended by the American Society for Microbiology (ASM).[67] The ASM method was proposed in the quality assurance plan considering that the Hanover Park raw sludge contained 60% by weight of biological sludge. The other two methods investigated were the freon-extraction method outlined in the APHA Standard Methods[58] for oil and grease and the chloroform-methanol extraction procedure suggested by O'Rourke[72] for digester feeds and effluents. The ASM and the O'Rourke procedures are semiwet methods utilizing chloroform-methanol extractions, and are probably not as accurate as the APHA Soxhlet extraction procedure. Since Soxhlet extraction is recommended for oil and grease, it had to be compared with the ASM and O'Rourke procedures to ascertain its capability to extract biological lipids.

Recovery of "Standard" Lipids--As a first step, the three selected lipid-determination methods were compared with respect to their abilities to recover "standard" or common lipids. Crisco™ was used as the common natural lipid. Motor oil was selected as the common synthetic lipid. The results reported in Table 20 show that Soxhlet extraction exhibited precision and accuracy that were comparable to those of the ASM and the O'Rourke methods. Also, it was evident that lipid recoveries by Soxhlet extraction with freon were not affected by the ratio of natural to synthetic lipids.

Recovery of Sludge Lipids--The three selected lipid-analysis procedures were also compared in terms of their efficiencies to recover sludge lipids. Analyses were conducted with sludge samples alone and with sludge samples mixed with known quantities of an external standard, motor oil. Standard recoveries were calculated from concentrations determined for sludge and the sludge-standard mixture. Results reported in Table 21 show that freon Soxhlet extraction was better than the other two methods in terms of biological lipids recovery. Also, freon extraction recovered the non-biological lipid with efficiencies comparable to those of the other two procedures.

Selected Lipid Analytical Method--Based on the analytical work described above, it was concluded that the ASM method involved a slow solvent evaporation rate and a lyophilization step. The O'Rourke method involves a slow filtration step. Both methods are time-inefficient and require the use of hazardous solvents. Quantitative lipid transfer from one step to another was a problem, and results from these semiwet methods do not compare well with those from the Soxhlet extraction procedure known to be more accurate for oils, grease, and waxes.

Although the freon Soxhlet extraction procedure is not clearly specified to be suitable for biological lipids, results of investigations reported above showed that this method afforded increased recovery of sludge lipids over the alternative techniques tested. It was felt that the Soxhlet method is simple, straightforward, time-efficient, and at least as precise and accurate as the other lipid-analysis methods; also, freon appears to be less hazardous than the other solvents required for the semiwet techniques. Last, but not least, the APHA procedure should be preferred to other methods because results from this research can then be compared with those of others. One possible disadvantage is that freon Soxhlet extraction may be more expensive than the other extractions. Overall, the APHA Soxhlet procedure seemed to be better than the ASM and O'Rourke methods, and it was selected for this project.

TABLE 20. RECOVERIES OF COMMON LIPIDS BY THE SOXHLET, ASM, AND O'ROURKE METHODS

		Soxhlet method		O'Rourke method		ASM method	
Sample	Standard lipid in sample	Extracted lipid, g	Recovery, %	Extracted lipid, g	Recovery, %	Extracted lipid, g	Recovery, %
1[*]	5 g Motor oil + 15 g Crisco	19.4774	97.4	--	--	--	--
	15 g Motor oil + 5 g Crisco	19.3826	96.7	--	--	--	--
2[†]	50 g Motor oil	--	--	48.5787	97.0	--	--
	50 g Motor oil	--	--	47.9892	96.0	--	--
3	5 g Motor oil	--	--	--	--	5.0601	101.2
	5 g Motor oil	--	--	--	--	5.0209	100.4

[*] Sample 1 could not be analyzed by the O'Rourke and the ASM methods because of quantitative lipid transfer problems.

[†] Sample 2 could not be analyzed by the ASM method because this procedure is designed for small samples.

TABLE 21. RECOVERIES OF SLUDGE LIPIDS AND MOTOR OIL BY THE SOXHLET, ASM, AND O'ROURKE METHODS

Analytical method	Raw sludge lipid conc.,* wt % of TS	Recovery of motor oil standard from mixed sludge-oil sample, %
Soxhlet freon extraction	a) 27.6	94.1
	b) 24.3	78.6
O'Rourke method (chloroform-methanol extraction)	17.2	94.1
ASM method (chloroform-methanol extraction)	16.5	—

* Analysis was run on raw sludge sample only.

ANAEROBIC DIGESTIBILITY POTENTIAL TEST (ADPT)

Test Concept

The theoretical digestibilty potential of the digester feed sludge can be calculated from its elemental analysis assuming stoichiometric conversion of the organics to product gases. This theoretical potential, however, cannot be achieved in practice because only a part of the organics (volatile solids) is anaerobically biodegradable. An anaerobic digestibility potential (ADP) test was conducted with the Hanover Park feed sludge to estimate the anaerobic biodegradability potential of this substrate by long-term batch digestion at a selected mesophilic reference temperature of 35°C. The final methane yield and VS reduction obtained from this test serve as "bench marks" against which other experimental yields and VS reductions can be compared to evaluate the efficacy of the particular digestion system.

The ADP test is based on a concept similar to that of the long-term BOD test; the final methane yield and VS reduction of the ADP test are anaerobic counterparts of the "ultimate" BOD.

ADP Test Protocol and Data Analysis Procedure

The ADP test is started with an appropriate inoculum (seed) and a selected volume of the test sludge to produce measurable volumes of gas during selected incubation periods. In this research, the inoculum was obtained from a single-stage high-rate digester, which was continuously fed with Hanover Park sludge at an HRT of 7 days and exhibited satisfactory and stable performance.

The test is set up in triplicate by filling 282-mL-capacity glass serum bottles with selected volumes of the digester feed sludge, the inoculum, and deoxygenated water to obtain a final culture volume equal to about one-half the volume of the bottle. All transfers were made anaerobically, and the serum bottle was purged with a 20%-CO_2-80%-N_2 gas mixture that was passed through a heated copper column to remove any oxygen from this purge gas. The bottle was stoppered, crimp-sealed, and incubated at 35°C in an inverted position to minimize gas leaks. Control digester bottles were also prepared in the same manner but without the test sludge to be able to correct for gas production from the inoculum sludge, and thus to obtain net gas production from the feed sludge only. Tests with feed and seed sludges were conducted in triplicate.

Gas production and composition were determined for all bottles at selected intervals; a computer program was utilized to perform the tedious calculations necessary to determine accumulative and corrected biogas and methane productions at various incubation times. All measured gas volumes were reduced to dry volumes under the standard conditions of 15.55°F and 762 mm Hg mercury pressure. The ADP test was continued until gas production leveled out and no further measurable gas production could be observed. At termination, the contents of the bottles were mixed, and TS and VS analyses were performed in triplicate on aliquots of digested residue sampled from the bottles. Total and volatile solids balances were performed to check if gases were lost during the test, and to estimate VS reduction achieved by long-term digestion.

ENZYMATIC PRETREATMENT OF SLUDGE

Enzyme pretreatment of sludge was conducted using cellulase-cellobiase, and lipase enzymes obtained from Novo Laboratories Inc. (Cellulast 1.5 L, Novozym 188, and Novozym 225, respectively). These enzymes were selected on the basis of work conducted by SYSTECH Corporation for the U.S. EPA.[73] Cellulase aids the breakdown of cellulosic substrates to glucose, cellobiose and higher glucose polymers; cellobiase was used to convert cellobiose, a non-fermentable carbohydrate, to glucose. Lipase was used to aid the hydrolysis of feed sludge lipids to volatile fatty acids. Samples of these enzymes were obtained from Novo Laboratories Inc. as liquid slurries; the TS contents of the cellulase, cellobiase, and lipase were 0.665, 0.495, and 0.300 g/mL, respectively.

Digester feed slurries were pretreated with cellulase and cellobiase for 24 hours in containers incubated at 35°C. Dosages of 2.76 g cellobiase TS/kg feed TS and 0.28 g cellobiase TS/kg feed TS were used; the feed slurry was adjusted to pH 5 with 2.5N HCl prior to pretreatment and back to the original slurry pH (about 5.5) with 2.5N NaOH after pretreatment. Lipase was dosed directly to the acid-phase digestion with a timer-operated peristaltic pump about 43 times per day at a dosage of 2.75 g lipase TS/kg feed TS. Lipase was added directly to the acid-phase digester, instead of to the pretreatment container because its activity is optimum at the pH of the acid-phase digester (pH 6 to pH 7) and is greatly reduced at the pH used for the cellulase/cellobiase pretreatment. The enzyme dosages used in this work were found to be optimum by SYSTECH Corporation.[73]

SYSTEM START-UP AND OPERATION

Culture Start-Up and Acclimation

As indicated previously, eight different digesters were used to conduct the various single-stage, separate acid-phase and two-phase digestion runs. Each of these digesters was initially started with active mesophilic inocula obtained from either ongoing bench-scale digesters operated at IGT or full-scale digesters operated at MSDGC's West-Southwest wastewater treatment plant in Stickney, Illinois. Prior to inoculation, each digester was filled with water to expel air and then drained under a gas purge containing 70 mol % methane and 30 mol % carbon dioxide. Gas purging was continued after the water was drained until all traces of oxygen in the digester were removed, as determined by periodic gas analyses. The inocula were then anaerobically transferred to the digesters.

The mesophilic cultures were then acclimated to Hanover Park feed sludge with daily feeding at HRT's of 15 to 20 days for the single-stage and methane-phase digesters and at HRT's of 6 to 7 days for the acid-phase digesters. The single-stage and methane-phase HRT's were gradually reduced to the target HRT and loading-rate conditions while gas composition and effluent volatile acids concentrations were monitored to ensure that the methanogenic populations were not washed out of these digesters. Acid-phase HRT's were reduced more rapidly to enrich the acidogenic populations in these digesters.

Thermophilic cultures were developed from acclimated mesophilic cultures by increasing the digester temperature to 55°C in a single step after a volume of feed sludge equal to about 10% of the culture volume was added. The single-stage and methane-phase thermophilic cultures were left in batch for several days until effluent volatile acids had decreased to acceptable levels before daily feeding was started. Daily feeding was started immediately for the thermophilic acid-phase cultures.

Frequency of Digester Feeding

As indicated in the experimental plan, various steady-state CFCSTR digestion runs were to be conducted at digester HRT's of between 15 and 0.8 days. Digesters operated at HRT's lower than 15 days were fed, in small slug doses, 12 to 40 times per day with the auto-feed systems described in a previous section. The feed frequency was increased as the digester HRT was reduced in consideration of the relationship between feed frequency and microbial growth rate. Digesters were fed manually once per day after a selected volume of digester contents was wasted (withdrawn) when the HRT was 15 days.

The frequency of digester feeding used in this research varied from once to about 40 times per day. As will be evident from the following theoretical considerations, the above feeding frequencies were selected so that intermittent or semicontinuous feeding mode was for all practical purposes equivalent to continuous feeding.

Theoretical Basis for Equivalency of Intermittent and Continuous Feeding

Intermittent or semicontinuous digester operation is characterized by regular withdrawal (or wasting) of a part of the spent medium (or digester content) after a selected time interval of digestion and the replacement of the part withdrawn by fresh substrate or digester feed slurry. When the number of withdrawals and feedings per unit time is infinity or sufficiently high, the semicontinuous or intermittent feeding converts to continuous feeding. Under steady-state conditions in a CFCSTR digester, a dynamic equilibrium is established between the increment of microbial mass in the digester grown at the expense of the slug feed and its decrease with effluent drawoff, so that the total mass of microorganisms in the digester after wasting is constant. At steady state the specific growth rate, μ, of the digester organisms is related to the dilution rate by Fencl's equation,[74] as follows:

$$1 = (1 - \frac{D}{n})^n \exp \mu \qquad (1)$$

where D = dilution rate = θ^{-1}

θ = theoretical detention time = V/F

V = digester volume

F = flow rate

n = number of digester wastings and feedings per unit time.

When n is infinity, the semicontinuous process passes into the continuous process, and Equation 1, in this case, can be rewritten as —

$$1 = \lim_{n \to \infty} \Sigma \exp \mu(1 - \frac{D}{\mu})^n \qquad (2)$$

The solution of Equation 2 is —

$$\mu_{CF} = D \qquad (3)$$

That is, for the "ideal" case of continuous feeding (CF) and withdrawal, the specific growth rate, μ_{CF}, is equal to the dilution rate.

Further analyses of Equations 1 and 3 show that, in theory, there is little difference between semicontinuous and continuous digestion provided θ and n are sufficiently large. As θ and n are decreased, the semicontinuous process deviates more and more from the ideal continuous process in that the specific growth rate of the organisms must be increasingly greater than the dilution rate if steady-state digester operation is to be achieved.

For example, the specific growth rate at steady state for a CFCSTR digester fed once per day (n = 1) at a 15-day HRT must be only 3% higher than the dilution rate (Figure 11). Thus, for all practical purposes, daily feeding once per day is equivalent to continuous feeding at a 15-day HRT.

However, for a steady-state CFCSTR digester operated at a 2-day HRT with daily feeding, the growth rate must be 39% higher than the dilution rate (Figure 12). As the feed frequency is increased to 15 times per day or more, the deviation between specific growth rate and dilution rate is reduced to less than 3%. For this reason, feed frequencies for digesters operated at HRT's of less than 15 days were selected so that the deviation between specific growth rate and dilution rate would be 3% or less.

pH Control

Culture pH's were controlled only for the parametric-effects acid-phase digestion runs because one of the objectives of these runs was to ascertain the effects of selected pH's (pH 5 and pH 7) on acidogenesis of sludge. The culture pH's were controlled continuously, with the apparatus described previously, by dosing the cultures with 2.5N NaOH (for pH 7) and 2.5N HCl (for pH's below 7). Sodium bicarbonate ($NaHCO_3$) and lime [$Ca(OH)_2$] were considered as pH control chemicals for the pH 7 runs but were rejected because bicarbonate could act as a carbon source for gas production, and lime is incapable of maintaining culture pH's above 6.8. Hydrochloric acid was used to control the cultures below pH 7 (instead of sulfuric or nitric acids) because the chloride ion is less toxic to anaerobes than the sulfate or nitrate ions. The normalities of the pH control chemical solutions were selected so that the volumes dosed for pH control were high enough to measure accurately, but low enough so as not to significantly dilute the cultures or affect the digester HRT's.

Several procedures were used to ensure that the pH controllers functioned properly. During the first 2 months of operation, chart recorders were attached to both controllers to provide a continuous record of pH; these charts were scanned particularly to check for large pH excursions that may have occurred during non-working hours. Analog pH meters incorporated in the controllers were checked several times per day. Effluent pH measurements were made at least three times per week with a bench-top pH meter and compared with the controller pH meter readings. In addition, the in-line pH probes for both controllers were calibrated at least once per week with standard buffer solutions.

During the first few weeks of operation with automatic pH control, several large pH excursions were observed. The problems were resolved by lowering the pH probes further into the cultures to improve contact with the culture and by moving the pH control delivery tubes closer to the pH probes to improve pH response time. Thereafter, the controllers functioned independently and maintained culture pH to within 0.15 pH units of the setpoint.

Foam and Scum Control

Floating scum and foam were encountered during mesophilic operation at short HRT's (less than 5 days) and high loadings (more than 10 kg VS/m^3-day).

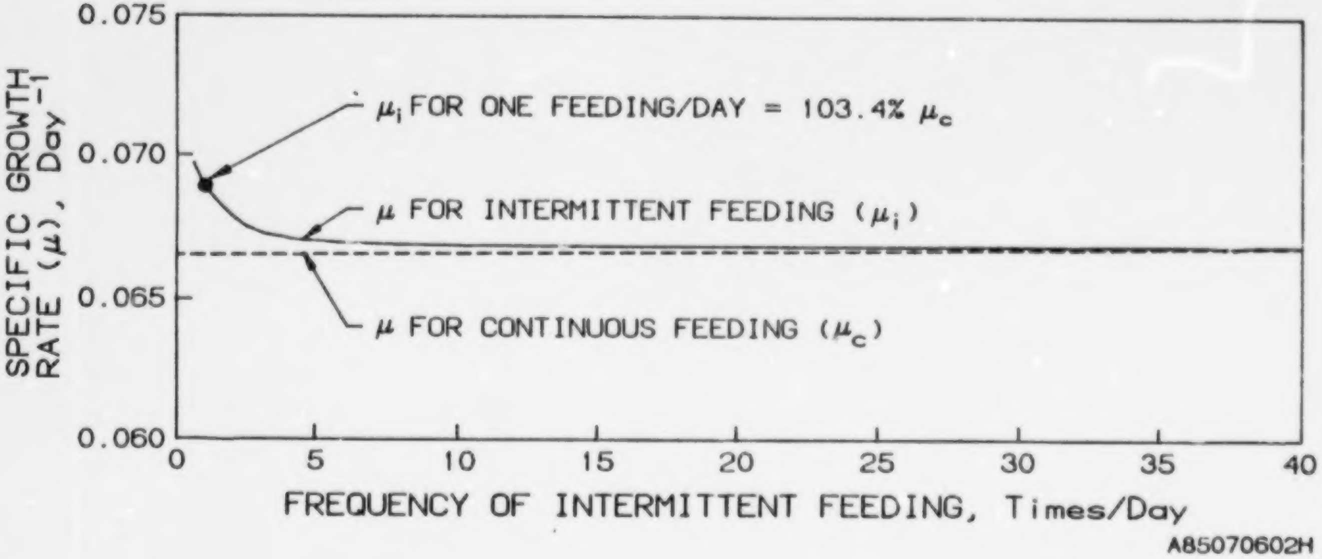

Figure 11. Effect of feeding frequency on specific growth rates in a CFCSTR digester operated at a 15-day HRT.

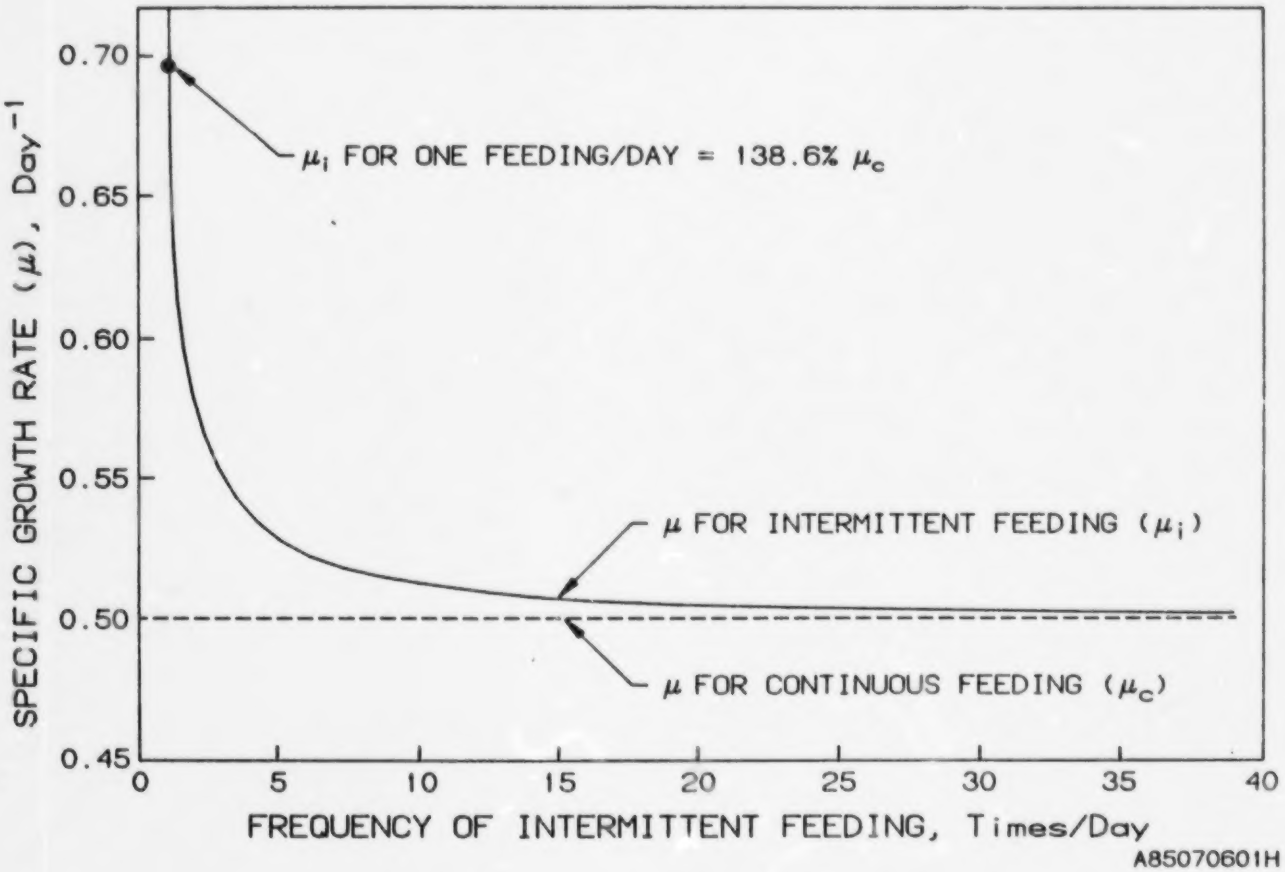

Figure 12. Effect of feeding frequency on specific growth rates in a CFCSTR digester operated at a 2-day HRT.

Scum and foam interfered with digester operation and gas collection; however, these problems were not observed in the thermophilic digesters. In many cases the scum or foam moved out of the digester with the product gases, fouling the gas collections tubings and valves. Dissolved gases and foam in the digester effluent resulted in loss of gas-liquid seals in the effluent overflows, which interfered with effluent withdrawal.

Several strategies were investigated to eliminate these problems. An antifoam agent (Dow-Corning FG-10) was added to the feed slurry and intermittent mixing was instituted instead of continuous mixing. Neither of these actions had any appreciable effect on scum formation, however. Foam traps were then installed in the gas lines between the digesters and the gas collection systems, and the effluent overflow systems were modified.

The foam traps consisted of 2-L vessels and were installed so that product gases from the digesters entered at the bottom and exited at the top of the vessels before passing to the gas collection systems. Thus, any foam carried out of the digesters with the product gases could be collected in the foam traps before it fouled the collectors. The traps were drained manually as necessary. The original 1.9 to 2.5-cm-diameter overflow pipes were replaced with larger 3.8 to 5.1-cm-diameter overflows to minimize gas locking caused by the foam and scum in the effluent. In two of the digesters (Digester Nos. 332 and 333), the overflow pipes were installed through the digester walls and 90° elbows were installed on the pipes inside the digesters. The open ends of the elbows were directed downward and were submerged in the culture to a depth of about 4 cm so that only effluent from beneath the foam and scum layer was withdrawn. A perforated plate was also mounted in Digester No. 333 just below the culture surface to keep the foam layer submerged and wetted. Although these modifications did not totally eliminate the accumulation of scum and foam within the digesters, they did resolve the problems associated with gas collection and effluent withdrawal.

Process Monitoring

Routine Monitoring--

Digestion runs were routinely monitored by determining digester HRT's, culture temperatures, gas production rates (GPR), gas composition, and effluent pH and volatile acids concentrations according to the schedule in Table 22. These data were regularly plotted and reviewed to assess the progress at each run and to determine when steady-state operation was achieved. Gas production rates were monitored daily because this determination was the easiest and most accurate means of tracking the performance of the runs and because it was the primary performance variable used for selecting steady-state segments of the runs. Gas compositions and effluent pH and volatile acids were measured less frequently because these parameters varied less with time.

Steady-State Monitoring--

The schedule for process monitoring during steady-state operation was similar to that followed during nonsteady-state operation (Table 22), except

TABLE 22. ROUTINE PROCESS MONITORING SCHEDULE

Determination	Non-steady-state frequency	Steady-state frequency
Digester HRT	Daily	Daily
Culture temperature	3 to 7 times per week	3 to 7 times per week
Digester gas production rate	Daily	Daily
Gas composition	1 to 2 times per week	2 to 4 times per week
Effluent pH	3 to 7 times per week	3 to 7 times per week
Effluent volatile acids	1 to 2 times per week	2 to 4 times per week

that gas compositions and effluent volatile acids were measured more frequently to ensure that these process variables were also stable with time. As described previously, feed and effluent slurry samples were also collected during steady-state operation and analyzed for solids, organic components, alkalinity, and nitrogen contents.

Steady-State Criteria--

This research required collection of steady-state data for most of the experimental phases. Steady-state was defined as a segment of a digestion run during which the digester operating variables and performance parameters were maintained at "constant" levels, permissible within the constraints of bench-scale equipment operability and the available measurement techniques and for which solids balances were between 85% and 115%. For complete-mix systems, the steady-state duration was equal to at least twice the HRT. The criterion used for achievement of steady state was the constancy of certain digester operating and performance data during a selected run segment. A particular parameter was assumed to have reached a constant level if the coefficient of variation was less than that specified in Table 23. Digester HRT's, loading rates, and culture temperatures were the parameters that defined the operating conditions of each run and were thus used for the operation criteria. The primary performance criteria were gas and methane productions, which were measured more accurately and frequently than the other performance parameters. Solids balances were also used as indicators of steady state because the solids analyses could be completed quickly, and because good mass balances are prerequisites for steady-state operation.

The levels of variabilities specified in Table 23 are reasonable in view of the unavoidable variabilities associated with control of the operating

TABLE 23. STEADY-STATE CRITERIA

Process variable/performance parameter	Maximum acceptable coefficient of variation, %
Operation	
HRT, days	20
Loading rate, lb VS/ft^3-day	20
Temperature, °C	5
pH*	5
Performance	
Methane content, mol %	10
Methane yield, SCF/lb VS added	20
Methane production rate, vol/day-culture vol	20

* This variable was a criterion for only the pH-controlled parametric-effects acid-phase digesters.

variables at selected levels and measurement of the performance parameters. For example, a 20% variability in sludge pumping rate is normal for the type of equipment available commercially. A variability of 20% in HRT is thus almost unavoidable. If feed sludge is delivered with a 20% variability in rate, gas yield and production rate would also have the same variability.

Data Reduction

The collected raw data were reduced to provide the operating and performance parameters indicated in Table 24. The reduced data were tabulated and graphed, as appropriate, for evaluation and interpretation of the experimental observations, and for arriving at conclusions with regard to parametric effects on digestion process efficiency. Data reduction also included the computation of such simple statistics as the mean, standard deviation, coefficient of variation, and correlation coefficient.

Hydraulic retention times and organic loadings were calculated based on daily measurements of feed slurry flow rates. Volatile solids concentrations used for the loadings were based on direct analyses of the feed slurries

TABLE 24. REDUCED OPERATING AND PERFORMANCE PARAMETERS

Analysis or measurement	Reduced data
Hydraulic retention time (HRT)	days
Organic loading rate	kg VS/m^3 culture-day
pH control chemical dosage	meq/L feed
Total solids (TS)	mg/L or wt %
Volatile solids (VS)	mg/L or wt % of TS
Fixed solids (FS)	mg/L or wt % of TS
Total suspended solids	mg/L or wt % of TS
Volatile suspended solids	mg/L or wt % of TS
Gas volume	Standard m^3 (dry) wt 15.55°C and 762 mm Hg
Total gas and methane yield	Standard m^3/kg VS added
Total gas and methane production rate	Standard vol/culture vol-day
Gas composition	Normalized mol %
Volatile fatty acids (VFA)	mg/L
Total VFA	mg/L as acetic
pH	Dimensionless unit or moles/l [H^+]
Total and bicarbonate alkalinities	mg/L as $CaCO_3$
Volatile fatty acids	mg/L as acetic acid
COD (total and filtrate)	mg/L or g COD/g VS
Nitrogen (ammonia)	mg/L NH_3-N or wt % of TS
Nitrogen (organic)	mg/L Org-N or wt % of TS
Crude protein	mg/L or wt % of VS
Lipids	mg/L or wt % of VS
Carbohydrate	mg/L or wt % of VS
Carbon and hydrogen	wt % of TS
Sulfur	wt % of dry solids
Phosphorous	wt % of TS
Heating value	kcal/kg (dry)
Feed component reduction	percent
Solids balances	percent

during steady-state runs. During nonsteady segments of runs, the VS concentrations were calculated on the basis of the concentrated feed solids analyses and the dilution factor used to prepare the slurry. Chemical dosages for the pH-controlled parametric-effects acid-phase runs were determined on the basis of the daily flow rates of chemical solutions and feed slurry.

Gas production volumes were converted to standard volumes at standard temperature and pressure (15.55°C and 762 mm Hg) on a dry basis using daily barometric pressure and ambient temperature readings taken in the digester laboratory. Gas yields were reported in units of standard m^3/kg VS added instead of standard m^3/kg VS reduced, sometimes used by other researchers, because the former units better describe the conversion efficiency of feed volatile solids to gas. Methane yields were calculated on a daily basis as the product of the total gas yield and the measured methane content; on days when gas composition was not analyzed the average of the previous and subsequent methane contents was used to calculate the methane yield. Steady-state methane yields were reported as the mean of these daily yields during the steady-state segment of each run. Daily and mean steady-state methane production rates were calculated in a similar manner. Analyzed gas compositions generally totaled less than 100%, primarily due to water vapor in the gas samples (which was not detected by the chromatograph) and experimental error. For these reasons, gas compositions were reported on a normalized basis; the individual gas components were multiplied by a constant factor to bring the total to 100.0%.

Feed and effluent slurry analyses were generally reported in units of mg/L to permit direct comparison of feed and effluent slurry characteristics. Organic component analyses (crude protein, carbohydrates, and lipids) were also reported as wt % of VS because these compounds were the major constituents of volatile solids in the feed and effluent slurries.

VS Reduction Efficiency

Organic component reductions were calculated as the percent ratio of the difference of feed and effluent concentrations to feed concentration. Volatile solids reductions can be calculated by three different methods, as follows:

Mop-16 Method

$$VS_R = \frac{VS_i - VS_o}{VS_i - (VS_i \times VS_o)} \times 100 \qquad (4)$$

Mass of Gas Method

$$VS_R = \frac{W_g}{VS_f} \times 100 \qquad (5)$$

Carbon-in-Gas and Carbon-in-Feed Method

$$VS_R = \frac{F_p \text{ X } C_g}{VS_f} \text{ X } 100 \qquad (6)$$

where VS_i = influent VS, decimal wt % of TS

VS_o = effluent VS, decimal wt % of TS

W_g = daily mass flow of product gases, g/day

VS_f = daily mass flow of feed VS, g/day

C_g = daily mass flow of carbon in product gases, g/day

F_p = correlation factor (1.84), g VS/g carbon

Equation 4 was used to calculate VS reduction because this method is recommended by the Water Pollution Control Federation.[2] This method of determining VS reduction is based on the assumption that the mass flow rates of fixed solids in the feed and effluent slurries are the same and it accounts for the changes in slurry volume which take place during digestion as a result of gas production. It is, however, sensitive to errors in the determination of the feed and particularly the effluent VS contents.

In general, errors in the determination of feed VS content are limited to normal analytical errors, whereas determinations of effluent VS contents are also subject to errors due to incomplete volatilization of the sample and differences in the fixed solids contents relative to those of the feeds. The physical/chemical characteristics of the effluent slurries are different than those of the feed slurry as a result of digestion. Inorganic films develop on dried effluent solids and shield the sample from complete volatilization. The organic residue in effluent slurries may also have different volatilities than the feed organic due to differences in composition, solids particle size, and porosity. In addition, effluent slurries usually have higher bicarbonate alkalinities than feed slurries due to conversion of organic carbon to inorganic forms, which results in a net increase in fixed solids content. These errors are further aggravated when external chemicals are added directly to the digester, as was the case for the pH-controlled and enzymatic pretreatment digestion runs.

For these reasons, two other methods of calculating VS reduction (Equations 5 and 6) were investigated. Both methods are based on the assumptions that the mass flow rates of feed VS and product gases can be accurately determined, and the problems associated with effluent VS determination can be avoided. In Equation 5, the mass rate of gas production is assumed to be equal to the rate of VS reduction (in other words, each gram of VS reduction is equal to 1 gram of gas production). Although this assumption seems reasonable, it is subject to potentially large errors because the weight ratio of product gas formed per unit VS reduced varies between 0.5 and 1.6 g gas/g VS converted, depending on the elemental composition of the reduced organic component.[2] Ratios greater than 1 occur when a portion of the hydrogen and oxygen in the methane and carbon dioxide product gases are contributed by water during hydrolysis of the organic component. For example, stoichiometric conversion of a carbohydrate with an assumed composition of $C_6H_{10}O_5$ results in the following balanced reaction:

$$C_6H_{10}O_5 + H_2O \; T \; 3CO_2 + 3CH_4$$

For this reaction, the weight ratio of product gas to carbohydrate VS reduced is 1.11. Similarly, the ratio for conversion of lipids is 1.6, assuming a composition of $C_{50}H_{90}O_6$. Ratios lower than 1 may occur for conversion of proteins because a portion of the carbon dioxide produced becomes chemically bound to ammonia (produced during decomposition of amino acid groups) to form ammonium bicarbonate. Thus, the weight ratio of product gas to VS reduced is dependent not only on the elemental composition of each organic component but also on reduction of each component.

Equation 6 was developed in an attempt to avoid the problems associated with hydrolysis, inherent in Equation 5. In this method, the mass flow rate of VS reduced is calculated as the product of the mass flow rate of product gas carbon and a proportionality factor relating carbon to equivalent VS. It was reasoned that carbon in the product gas could be produced only from conversion of feed VS carbon. Thus, if a correlation between feed VS and carbon content could be established, the mass rate of VS reduced could be calculated based on gas production and composition data. The correlation factor used (1.84 g VS/g carbon) was determined on the basis of a linear regression analysis of carbon and VS contents in 10 sewage sludge feeds and 4 effluent slurries (Figure 13). Volatile solids and carbon contents, which form the basis for Figure 13, and the correlation analysis are detailed in Table 25. The correlation coefficient for all 14 data sets was about 99% indicating a strong correlation between carbon and VS in both feeds and effluents.

As a further check on the MOP-16 method, the data were examined to see if concentration of ash (mg/L TS-mg/L VS) was the same in the feed and effluents from the digesters. This calculation (see Table G-1) showed losses relative to the total feed solids to be low, about ±2 percent. Surprisingly, these small differences caused large discrepancies between VS_R's (volatile solids reduction) calculated by the MOP-16 method and the material balance method. The absolute differences ranged from -7 to 10% (see Table G-1). The methods gave the same result when there was no ash loss. As is shown below, the results by the two methods correlated better with each other than with results of the mass-of-gas method:

Comparison	Correlation Coefficient (r)
MOP-16 vs. Material Balance	0.84
Mass of Gas vs. MOP-16	0.70
Mass of Gas vs. Material Balance	0.78

The conclusions of the report would not be substantially changed if any one of these three methods were used to calculate VS_R. However, when the inaccuracies in determining true volatile solids levels in the effluent were considered, the mass-of-gas method appeared to be the best choice.

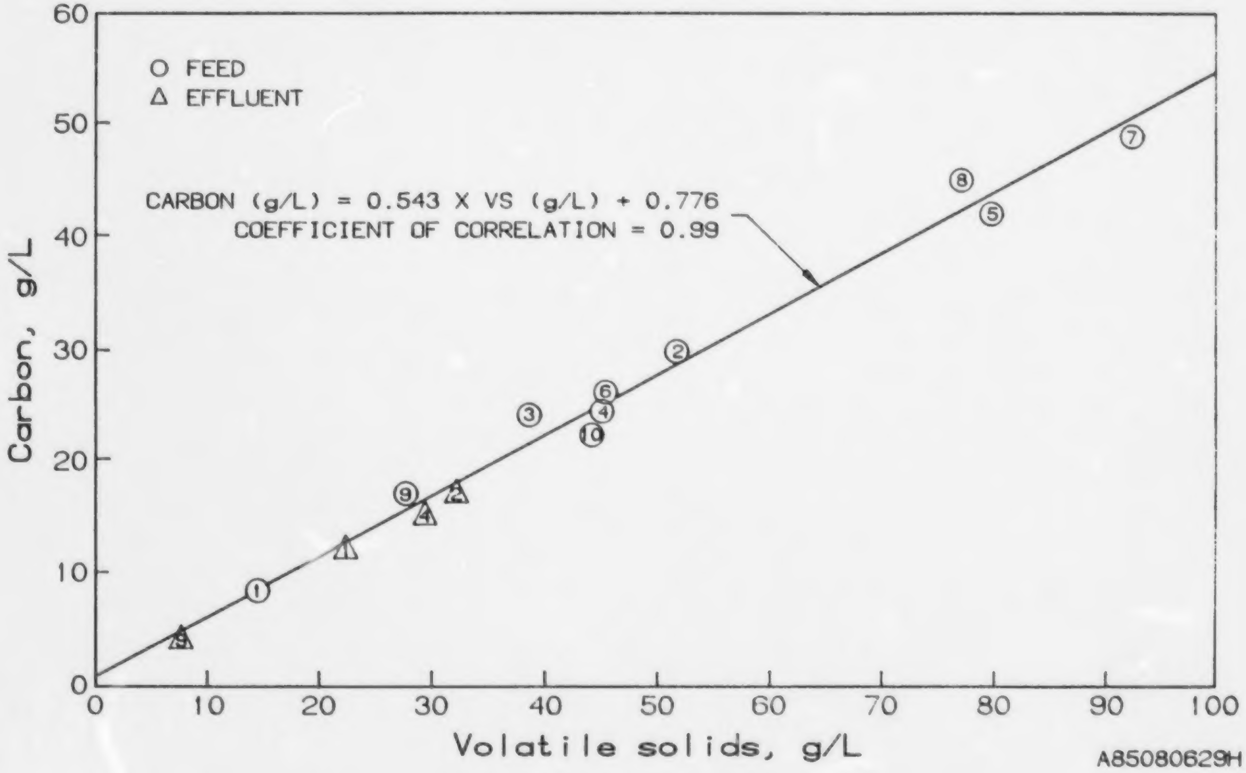

Figure 13. Correlation between carbon and volatile solids concentrations of raw and digested sewage sludges.

TABLE 25. VOLATILE SOLIDS AND CARBON CONTENTS OF RAW AND DIGESTED SEWAGE SLUDGES

		Raw sludge		Digested sludge	
Sample no.	Source of sludge	Volatile solids, S/L	Carbon, g/L	Volatile solids, S/L	Carbon, g/L
1	Hanover Park mixed primary/activated	14.66	8.33	22.43	12.36
2	Hanover Park mixed primary/activated	51.73	30.01	32.09	17.44
3	SESD primary (Salem, MA)	38.64	24.41	--	--
4	Hanover Park mixed primary/activated	45.10	24.86	29.42	15.44
5	Stickney activated	79.24	42.28	--	--
6	Hanover Park mixed primary/activated	45.07	26.41	--	--
7	Stickney activated	92.06	49.20	--	--
8	Hanover Park mixed primary/activated	77.02	45.29	--	--
9	Disney World primary (Orlando, FL)	27.84	17.31	7.35	4.35
10	Stickney activated	43.98	22.64	--	--

SECTION 7

CHEMICAL CHARACTERIZATION OF PROCESS FEEDS

CHEMICAL CHARACTERIZATION OF UNPROCESSED RAW SLUDGES

Since raw sludges collected from the wastewater treatment plants were not directly fed to the digesters, limited chemical analyses were conducted to characterize them. More detailed chemical analyses were performed on the digester feed sludges which were prepared by processing the collected raw sludge.

The raw sludges were analyzed for TS and VS contents as reported in Tables 26, 27, and 28. The mixed primary-activated raw sludge from Hanover Park had VS contents between 68% and 76% depending on the season (Table 26). The primary raw sludge from Downers Grove had VS contents between 76% and 80% (Table 27). The activated sludge from Stickney had VS contents between 66% and 67% (Table 28). The mixed primary-activated and activated raw sludges were analyzed for total carbon, hydrogen, total sulfur, total nitrogen, total phosphorous, and heating (or calorific) value; the results of these analyses are reported in Table 29. Examination of the data in Table 29 showed that higher carbon and VS contents of the sludge solids gave rise to higher heating values, as expected. It is also evident that a unit mass of activated sludge VS had lower carbon content and calorific value than those of a unit mass of mixed primary-activated sludge VS; these observations indicated that the gas and methane yield potentials per unit mass of VS would also be lower for activated sludge.

The C/N and C/P ratios of the Hanover Park sludge were 8.3:1 and 28.5:1, respectively. From these ratios, it was concluded that this sludge was not deficient in nitrogen and phosphorus.

CHEMICAL CHARACTERIZATION OF DIGESTER FEED SLUDGE

Solids Analyses

Digester feed slurries were analyzed for total solids, volatile solids, and fixed solids during the steady-state and non-steady-state operating periods of the digestion runs. These data were reported in detail in Table A-1. Efforts were made to maintain the feed solids concentrations at levels specified under the experimental-design operating conditions outlined in Tables 7 through 9 (Section 5). However, deviations from these desired concentrations were unavoidable.

TABLE 26. COLLECTION, PROCESSING, AND SOLIDS ANALYSES OF HANOVER PARK RAW SLUDGE

Collected raw sludge							Processed raw sludge analyses[a]	
Date lot collected	Lot no.	Quantity, liters	TS, wt %	VS, wt % of TS	Batch no.	Processing method	TS, wt %	VS, wt % of TS
11/82	1	1400	2.00	73.47	1	none	2.00	73.47
					2	FTB[b]	4.99	73.13
					3	FTB	4.56	74.08
					4	FTB	11.98	59.74
					5	FTB	4.51	74.27
					6	FTB	3.65	74.47
					7	FTB	3.62	73.39
					8	FTD[c]	4.37	72.68
					9	FTD	4.91	74.25
					10	FTD	4.54	73.54
					11	FTD	4.95	64.56
					12	FTD	5.83	72.91
					13	FTD	4.82	73.14
					14	FTD	5.10	72.36
					15	FTD	5.13	73.16
12/82	2	1200	--	--	1	FTD	8.03	75.11
					2	FTD	8.64	73.99
					3	FTD	6.89	73.76
					4	FTD	9.82	75.40
4/83	3	1400	--	--	1	FTD	11.39	70.47
					2	FTDB	6.86	69.97
6/83	4	1200	--	--	1	FTD	7.70	72.25
					2	FTD	8.08	71.76
7/83	5	3000	--	--	1	FTD	6.09	64.61
					2	FTD	10.48	71.08
					3	FTD	12.30	69.54
					4	FTD	11.62	70.00
11/83	6	3600	--	--	1	FTD	9.31	68.10
					2	FTD	9.88	68.29
					3	FTD	14.11	68.45
					4	FTD	12.96	68.97

(continued)

TABLE 26 (continued)

Collected raw sludge							Processed raw sludge analyses[a]	
Date lot collected	Lot no.	Quantity, liters	TS, wt %	VS, wt % of TS	Batch no.	Processing method	TS, wt %	VS, wt % of TS
1/84	7	3600	--	--	1	FTD	5.96	76.38
					2	FTD	7.02	75.87
					3	FTD	7.77	74.15
					4	FTD	6.88	75.94
2/84	8	1400	3.07	76.80	1	LC[d]	11.05	78.58
					2	LC	6.26	78.13
					3	LC	6.14	74.57
					4	FTD	8.01	78.22
					5	FTD	5.15	77.34
3/84	9	3600	3.49	76.88	1	FTB	6.42	76.14
					2	FTB	5.99	75.73
4/84	10	600	2.04	67.98	1	FTD	7.25	68.86
					2	FTD	6.60	76.66
4/84	11	600	--	--	1	FTD	10.29	77.06
5/84	12	900	--	--	1	FTD	8.58	76.35
5/84	13	600	--	--	1	FTD	9.63	76.62
6/84	14	1200	--	--	1	PC[e]	8.47	71.36
6/84	15	600	--	--	1	PC	9.00	69.41
7/84	16	600	--	--	1	FTD	8.97	74.56
8/84	17	600	--	--	1	FTD	13.15	74.78

(continued)

TABLE 26 (continued)

Collected raw sludge							Processed raw sludge analyses[a]	
Date lot collected	Lot no.	Quantity, liters	TS, wt %	VS, wt % of TS	Batch no.	Processing method	TS, wt %	VS, wt % of TS
9/84	18	1400	--	--	1	FTD	13.76	72.92
					2	FTD	15.01	73.40
					3	FTD	21.06	72.23
					4	FTD	15.51	73.72
					5	FTD	16.01	50.51
					6	FTD	14.87	72.02
					7	FTD	15.35	72.29
					8	FTD	17.36	72.22
					9	FTD	17.34	72.16
11/84	19	1400	--	--	1	FTD	14.61	70.56
					2	FTD	14.66	71.22
					3	FTD	16.20	71.16
					4	FTD	13.29	70.76
					5	FTD	15.68	60.32
					6	FTD	13.98	71.54

[a] These analyses were performed immediately after preparation of the sludge batches and were used as guides to prepare digester feed slurries for the experimental runs. Separate solids analyses were performed on the digester feed slurries.

[b] FTB refers to raw sludge processing by freezing and thawing in 10-liter plastic bags (Method 1 in text).

[c] FTD refers to raw sludge processing by freezing and thawing in 200-liter drums (Method 1 in text).

[d] LC refers to raw sludge processing by laboratory centrifuge (Method 3 in text).

[e] PC refers to raw sludge processing by pilot centrifuge (Method 4 in text).

TABLE 27. COLLECTION, PROCESSING, AND SOLIDS ANALYSES OF DOWNERS GROVE RAW PRIMARY SLUDGE

Collected raw sludge							Processed raw sludge analyses	
Date lot collected	Lot no.	Quantity, liters	TS, wt %	VS, wt % of TS	Batch no.	Processing method	TS, wt %	VS, wt % of TS
10/84	22	50	4.90	79.67	1	homogenize	4.90	79.67
12/84	23	58	3.78	75.86	1	homogenize	3.78	75.86
12/84	24	15	4.63	77.82	1	homogenize	4.63	66.82
12/84	25	18	4.34	76.71	1	homogenize	4.34	76.71
12/84	26	20	4.26	77.51	1	homogenize	4.26	66.51

Table 28. COLLECTION, PROCESSING, AND SOLIDS ANALYSES OF STICKNEY RAW ACTIVATED SLUDGE

Collected raw sludge							Processed raw sludge analyses[a]	
Date lot collected	Lot no.	Quantity, liters	TS, wt %	VS, wt % of TS	Batch no.	Processing method	TS, wt %	VS, wt % of TS
9/84	20	870	13.67	65.68	1	D&B*	9.03	65.68
11/84	21	490	13.67	66.55	1	D&B	10.25	66.55

* D&B refers to raw sludge cake processing by dilution and blending.

Table 29. CHEMICAL CHARACTERISTICS OF UNPROCESSED RAW SLUDGES

	Mixed primary-activated sludge from Hanover Park (Lot 1)	Activated sludge from Stickney* (Lot 20)
Total carbon, wt % of TS	41.65	37.60
Total carbon, wt % of VS	56.91	51.46
Hydrogen, wt % of TS	6.25	5.68
Total sulfur, wt % of TS	1.50	0.85
TKN, wt % of TS	4.99	6.47
Total phosphorus, wt % of TS	1.46	2.00
Higher heating value		
Btu/lb TS	7,937	7,582
Btu/lb VS	10,846	10,378

* The Stickney activated sludge was mixed with Downers Grove primary sludge to prepare the digester feed slurries.

Selected digester feed slurries were also analyzed for total, volatile, and fixed suspended solids (TSS, VSS and FSS). These analyses are reported in Table A-2. A summary of these analyses is presented in Table 30. About 78% to 92 wt % of the total solids was insoluble or particulate matter with the balance being soluble. Of the total residue (i.e., total solids upon evaporation), 0.3% to 13.4 wt % was soluble inorganics (soluble fixed solids), about 12% to 32 wt % insoluble inorganics, about 2% to 11 wt % soluble organics (soluble VS), and 56% to 67 wt % insoluble organics. The average contents of soluble inorganics, insoluble inorganics, soluble organics, and insoluble organics were about 8.3, 21.4, 7.5, and 63.8 wt % TS, respectively. The data also indicated that on the average, about 89 wt % of the sludge organics were insoluble particulate matter. Direct measurement of suspended and dissolved total and volatile solids indicated that about 89 wt % of VS was insoluble and 11 wt % of the organics was soluble (Table 31). The information developed above from the solids analyses clearly indicated that the sludge feed was predominantly insoluble in nature, and hydrolysis of the particulate organics was an important consideration in gasification and stabilization of this substrate.

TABLE 30. SOLIDS ANALYSES OF DIGESTER FEED SLURRIES

Digester feed slurry prepared from feed lot/batch no(s)	Total residue, mg/L			Total suspended matter		Volatile suspended matter		Fixed suspended matter		Total soluble matter*		Volatile soluble matter*		Fixed soluble matter*	
	Total solids	Volatile solids	Fixed solids	mg/L	wt % of TS	mg/L	wt % of TS	mg/L	wt % of TS	mg/L	wt % of TS	mg/L	wt % of TS	mg/L	wt % of TS
5/3	71,600	50,280	21,320	65,740	91.8	44,710	62.4	21,030	29.4	5860	8.2	5570	7.8	290	0.41
5/4	40,280	28,660	11,620	33,560	83.3	27,100	67.3	6460	16.0	6720	16.7	1560	3.9	5160	12.8
5/4	39,350	28,100	11,250	32,100	81.6	25,910	65.8	6190	15.7	7250	18.4	2190	5.6	5060	12.9
5/4	74,380	48,780	25,600	65,160	87.6	41,800	56.2	23,360	31.4	9220	12.4	6980	9.4	8240	3.0
5/4	77,710	52,620	25,090	69,890	89.9	45,000	57.9	24,890	32.0	7820	10.1	7620	9.8	200	0.25
6/2	80,340	53,580	26,760	63,650	79.2	47,670	59.3	15,980	19.9	16,690	20.8	6580	8.2	10,780	13.4
6/2	69,460	46,930	22,530	62,170	89.5	45,690	65.8	16,480	23.7	7290	10.5	1240	1.8	6050	8.7
8/5	59,380	44,960	14,420	46,140	77.7	39,200	66.0	6940	11.7	13,240	22.3	5760	7.7	7480	12.6
8/5	55,960	42,890	13,070	43,740	78.2	36,700	65.6	7040	12.6	12,770	21.8	6190	11.1	6030	10.8
			Means		84.4		63.8		21.4		15.7		7.5		8.3

* Total, volatile, and fixed soluble matter contents of the feed slurries were determined by difference.

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TABLE 31. DIRECT MEASUREMENTS OF TOTAL, SUSPENDED, AND DISSOLVED SOLIDS CONTENTS OF DIGESTER FEED SLURRIES

Digester feed slurry prepared from feed lot/batch (no)s	Total solids (TS), mg/L	Volatile solids (VS), mg/L	Suspended solids (SS), mg/L		Dissolved solids (DS), mg/L		SS + DS*, mg/L		Volatile solids, % of total	
			Total	Volatile	Total	Volatile	Total	Volatile	Insoluble	Soluble
6/2	80,340	53,580	63,650	47,670	15,070	5,990	78,720	53,660	88.8	11.2
6/2	69,460	46,930	62,170	45,690	4980	5900	67,150	51,590	88.6	11.4

* Data reported in these columns compare favorably with the TS and VS data obtained independently in separate tests.

Elemental Analyses and Calorific Value

Elemental analyses and calorific value determinations were performed on selected digester feed slurries; the results of these analyses are reported in Table 32. Given the nature of the material sampled and the difficulty of collecting a "representative" sample, the accuracy of the data in Table 32 was satisfactory. Also, comparing the analyses performed on samples collected over a period of eight days, there seemed to be no evidence of decomposition of the feed sludge during storage, as discussed in the next section.

From a comparison of the elemental and calorific-value analyses of the raw and the digester feed sludge, it appears that the latter showed higher carbon analysis and heating value and lower nitrogen and phosphorus concentrations than those of the former. Consequently, the C/N and C/P ratios of the digester feed sludge were slightly higher than those of the raw sludge. It was speculated from the above observations that during sludge processing by freezing, thawing, and decanting and discarding liquids, relatively more soluble nitrogen and phosphorus than carbon were lost during the sludge concentration process.

Chemical Oxygen Demand (COD) Analysis

Samples of digester feed slurries were also analyzed for total and filtrate COD's, so that the data could be used to estimate theoretical methane yield. Results of the COD analyses for several feed sludges are reported in Table A-4. Table 33 shows that the total COD contents of Lots 5 and 6 Hanover Park sludges ranged between about 1.3 and 1.6 g COD/g VS and averaged at 1.4 g COD/g VS. About 93% of the sludge COD was due to particulate organic matter and 7% due to soluble organics.

The COD of Lot 8 feed sludge was considerably higher than most samples from Lots 5 and 6 sludges. About 91% of the COD was due to particulate organics and 9% due to solubles.

Overall, the COD data indicated that 91.93 wt % of the sludge organics was particulate matter, and this observation is in close agreement with that made on the bases of suspended solids analyses showing that 90% of the sludge VS was insoluble material.

Ammonia and Organic Nitrogens

Nitrogens present in sewage sludges are mainly found as ammonia and organic nitrogens; nitrite and nitrate nitrogens are also present, but the concentrations of these nitrogenous species are minor relative to those of ammonia and organic nitrogens. Digester feed sludges were analyzed for ammonia and organic nitrogens because these data are useful in assessing 1) the physical nature of the nitrogenous material, 2) the immediate and potential availability of nitrogen, 3) the protein content of the sludge, 4) the potential buffering capacity that can be generated during digestion, and 5) the degree of liquefaction taking place under various fermentation conditions.

TABLE 32. ELEMENTAL ANALYSES AND CALORIFIC VALUES OF DIGESTER FEED SLURRIES PREPARED FROM HANOVER PARK SLUDGE*

Sample date	Sludge lot/batch nos.	Elemental analysis, wt % of TS					Higher heating value	
		Total carbon	Hydrogen	TKN	Total sulfur	Total phosphorus	Btu/lb TS	Btu/lb VS
1/6/83	1/2	--	--	4.99 (0.62)†	--	--	--	--
1/18/83	1/3	--	--	3.82 (0.78)	--	--	--	--
7/25/83	4/2	43.05	6.71	4.03	1.19	--	8556	11,719
7/27/83	4/2	43.01	6.79	--	1.19	--	8685	11,612
7/29/83	4/2	42.54	6.54	--	1.29	--	8414	11,386
8/2/83	4/2	44.11	6.63	--	1.28	1.22	8613	11,655

* The samples were collected directly from a refrigerated and mixed feed reservoir.

† Number in parenthesis is NH_3-N; organic nitrogen is (TKN)-(NH_3-N).

TABLE 33. CHEMICAL OXYGEN DEMAND ANALYSES FOR DIGESTER SLURRIES

Digester feed slurry prepared from feed lot/batch no(s)	Volatile solids	Total COD		Filtrate (soluble) COD		Particulate COD (by diff)		Soluble COD	Particulate COD
	mg/L	mg/L	g/g VS	mg/L	g/g VS	mg/L	g/g VS	% of total COD	
5/1	48,795	--	--	6390	0.130	--	--	--	--
5/3	50,160	--	--	4922	0.098	--	--	--	--
5/3	50,160	79,530	1.585	4696	0.093	74,834	1.492	5.9	94.1
5/3	47,430	66,723	1.406	5673	0.119	61,050	1.287	8.5	91.5
5/3	53,440	87,096	1.629	4918	0.092	82,178	1.538	5.6	94.4
5/3	53,440	--	--	4979	0.093	--	--	--	--
5/3	50,280	78,047	1.552	6547	0.130	71,500	1.422	8.4	91.6
5/4	28,660	39,580	1.381	2745	0.095	36,835	1.285	6.9	93.1
5/4	28,100	34,822	1.239	2601	0.092	32,221	1.147	7.5	92.5
5/4	48,780	78,798	1.615	--	--	--	--	--	--
6/2	53,580	80,617	1.504	4211	0.078	76,406	1.426	5.2	94.8
6/2	46,930	77,702	1.655	4605	0.098	73,097	1.558	5.9	94.1
		Means	1.507		0.102		1.394	6.8	93.2
8/5	47,940	85,983	1.793	7968	0.166	78,015	1.627	9.3	90.7
8/5	44,960	81,896	1.821	7753	0.172	74,143	1.649	9.5	90.5
8/5	42,890	76,394	1.781	7042	0.164	69,352	1.617	9.2	90.8
		Means	1.798		0.167		1.631	9.3	90.7

The ammonia and organic nitrogen analyses for a number of digester feed slurries are presented in Table 34. The results of these analyses showed that the ammonia-nitrogen (expressed as N) concentration of the raw feed slurries ranged between about 220 to 620 mg/L (0.33 to 1.05 wt % of TS) depending on the sludge lot. Lot 5 had the lowest ammonia nitrogen concentration and Lot 16 the highest. However, even the lowest observed ammonia-nitrogen concentration was adequate for anaerobic metabolism.

The organic nitrogen concentration, which is a measure of particulate proteinaceous material, ranged from about 1900 to 2900 mg/L (3.0 to 4.5 wt % of TS) depending on the lot of raw sludge of the total nitrogenous material (as measured by TKN) present in raw sludge Lots 1, 5, 6, 12, 13, 14, and 28, 9 to 15 wt % was soluble and 98 to 91 wt % was particulate proteinaceous material that had to be hydrolyzed. Raw sludge Lots 8, 16, and 17 had higher concentrations (18 to 20 wt %) of soluble nitrogenous materials and consequently, lower concentrations of nitrogenous particulates. That raw sludge Lot 8 had higher soluble organics than Lots 1, 5, 6, 12-14, and 28 was also evident from considerations of COD and SS data analyses as presented in Tables 30 and 33.

Acid-Base Characteristics

The pH values and alkalinities of the raw feed sludges are summarized in detail in Table A-3 and summarized in Table 35. These analyses were conducted to delineate the acid-base characteristics of these substrates, and to assess the type of buffer capacities that would be generated during digestion.

Table 35 indicates that all sludges were acidic in nature with pH values usually less than 6.5. The bicarbonate alkalinities were between 2160 and 5513 mg/L as $CaCO_3$ which are regarded as satisfactory for digester feeds. It was expected that additional alkalinities would be generated during the digestion process to produce a high natural buffering capacity within the digester.

About 20% to 40% of the bicarbonate alkalinity was due to ammonium bicarbonate, except that for raw sludge Lots 8, 16, and 17 about 50% to 80% of bicarbonate alkalinity was due to ammonium bicarbonate (Table 35), the reason being that these three sludge lots contained much larger concentrations of ammonium compared to the concentrations in the other sludges. Interestingly, sludges having higher ammonium-bicarbonate alkalinities also exhibited higher volatile-acids-salts alkalinities. It may be speculated from these observations that sludge Lots 8, 16, and 17 contained particulates that were more readily liquefied than those of the other sludges.

Crude-Protein, Carbohydrate and Lipid Analyses

Hanover Park Sludge--

Feed slurries used to operate the sludge digesters were analyzed for the three major organic components — crude protein, total carbohydrate, and lipids. Generally, the samples were collected from the feed reservoir during a steady-state operating period. In some cases feed slurry samples were also

TABLE 34. AMMONIA AND ORGANIC NITROGEN CONTENTS OF DIGESTER FEED SLURRIES

Digester feed slurry prepared from raw sludge lot/batch nos.	Total solids	Totak Kjeldahl nitrogen, (TKN)		Ammonian nitrogen			Organic nitrogen		
	mg/l	mg/L	wt % of TS	mg/L	wt % of TKN	wt % of TS	mg/L	wt % of TKN	wt % of TS
1/8	45,100	2251	4.99	280	12.4	0.62	1971	87.6	4.37
4/2	64,390	--	--	569	--	0.88	--	--	--
4/2	45,810	--	--	385	--	0.84	--	--	--
5/1	69,635	2599	3.72	247	9.5	0.35	2352	90.5	3.37
5/3	67,420	2380	3.52	224	9.4	0.33	2156	90.6	3.19
5/3	77,930	2544	3.26	299	11.7	0.38	2245	88.3	2.88
5/3	71,600	2366	3.30	268	11.3	0.37	2098	88.7	2.93
5/4	39,350	1587	4.03	131	8.3	0.33	1456	91.7	3.70
5/4	82,170	2688	3.26	168	6.3	0.20	2520	93.8	3.06
			Means	223	9.4	0.33	2138	90.6	3.18
6/2	80,340	2701	--	354	13.1	0.44	2347	86.9	2.92
6/2	69,460	2263	--	219	9.7	0.31	2044	90.3	2.94
			Means	287	11.4	0.38	2196	88.6	2.93
8/5	63,020	3206	5.08	589	18.4	0.93	2617	81.6	4.15
8/5	59,380	3187	5.36	624	19.6	1.05	2563	80.4	4.31
8/5	55,960	2929	5.22	509	17.4	0.90	2420	82.6	4.32
			Means	574	18.5	0.96	2533	81.5	4.26
12/1	41,560	2104	5.06	237	11.3	0.57	1867	88.7	4.49
13/1	69,870	3120	4.45	327	10.5	0.46	2793	89.5	3.99
13/1	67,530	3122	4.61	302	9.7	0.44	2820	90.3	4.17
13/1	65,310	3255	4.97	337	10.4	0.51	2918	89.6	4.46
13/1	67,530	3254	4.81	321	9.9	0.47	2933	90.1	4.34
			Means	322	10.1	0.47	2866	89.9	4.24
14/1	68,440	2704	3.94	369	13.6	0.53	2335	86.4	3.41
16/1	67,250	3337	4.95	587	17.6	0.87	2750	82.4	4.08
16/1	66,735	3534	5.29	755	21.4	1.13	2779	78.6	4.16
16/1	46,045	2440	5.29	528	21.6	1.14	1912	78.4	4.15
			Means	623	20.2	1.05	2480	79.8	4.13
17/1	66,540	3032	4.55	582	19.2	0.87	2450	80.8	3.68
17/1	68,960	3067	4.43	585	19.1	0.84	2482	80.9	3.59
17/1	66,430	3208	4.82	546	17.0	0.82	2662	83.0	4.00
			Means	571	18.4	0.84	2531	81.6	3.76
28/1	66,840	3411	5.10	535	15.7	0.80	2876	84.3	4.30

TABLE 35. ACID-BASE CHARACTERISTICS OF DIGESTER FEED SLURRIES

Digester feed slurry prepared from raw sludge lot no.	pH	Ammonia nitrogen, mg/L	Alkalinity, mg/L as $CaCO_3$				Ammonium alk./total bicarbonate alkalinity, %
			Ammonium	Total bicarbonate	Volatile acids alk.	Total	
1	6.74	280	1000	3971	671	4642	25.2
4	--	477	1704	--	--	--	--
5	6.4	223	796	4426	857	5283	18.0
6	6.8	287	1025	3783	579	4362	27.1
8	6.7	574	2050	2493	2224	4717	82.2
12	5.9	237	846	2160	1090	3250	39.2
13	5.9	414	1479	3850	1783	5633	38.4
14	6.2	369	1318	5513	1662	7175	23.9
16	6.4	623	2225	2975	2883	5858	74.8
17	6.2	571	2039	3919	1798	5717	52.0
28	6.1	535	1911	--	--	4750	--

collected during the nonsteady-state operating period. Results of these analyses for the different feed slurries are reported in detail in Table A-6. A summary of these analyses is presented in Table 36.

The crude protein content of the Hanover Park sludge varied between a low analysis of about 27 wt % of VS to a high value of about 37 wt % VS (Table 36). Similarly, the total carbohydrate and lipid contents varied between 18 and 30 wt % of VS, and 20 and 38 wt % of VS. The average protein, carbohydrate, and lipids contents of the feed slurries used during two years of research were 32.5, 23.7, and 27.0 wt % of VS, respectively. Thus, crude protein was the largest organic component and carbohydrate the lowest. Taken together protein, carbohydrate and lipids accounted for 75 to 92 wt % of the volatile solids or total organics depending on the time of sludge collection; on the average, about 83 wt % of the organics could be accounted for by protein, carbohydrate, and lipids. Short-chain fatty acids and alcohols could form a significant part of the total organics. As shown in Table 36, 2 to 11 wt % of the feed sludge VS was accounted for by short-chain fatty acids which are not detected as carbohydrates, lipids, or protein. Thus, the analyzed organics constituted about 88 wt % of the volatile solids, and 12 wt % of the VS was unidentified organics. By comparison, Buswell and Neave reported that lipids, carbohydrates and protein accounted for about 91 wt % of raw sludge volatile solids.[75]

Inspection of the data shows that the protein, carbohydrate, and lipid contents of the sludge varied from month to month, but there was no evidence of any definitive type of cyclical variation.

Mixed Downers Grove Primary and Stickney Activated Sludge--

The sum total of the contents of crude protein, total carbohydrate, and lipids of the mixed Downers Grove primary and Stickney activated (DGPSA) sludges was the same as that of the Hanover Park sludge. However, the DGPSA sludge had a significantly higher protein content and a much lower lipid content than those of the Hanover Park sludge; both sludges had about the same carbohydrate content.

TABLE 36. CRUDE PROTEIN, TOTAL CARBOHYDRATE, AND LIPIDS ANALYSES OF DIGESTER FEED SLURRIES PREPARED FROM HANOVER PARK SLUDGE

Digester feed prepared from sludge lot/batch nos.	Organic component, wt % of VS*					
	Date sludge collected	Crude protein	Total carbohydrate	Lipids	Short-chain fatty acids + ethanol†	Total of proetein, carbohydrate, lipids, vol. acids and ethanol
1/8	11/82	36.8	19.5	23.1	2.33	81.7
3/2	4/83	—	—	24.5	—	—
4/1	6/83	—	—	31.5	—	—
4/2	6/83	—	—	32.5	—	—
5/1	7/83	30.2	—	—	—	—
5/3	7/83	26.9	26.5	27.4	2.50	83.3
5/4	7/83	30.8	29.6	24.3	1.94	86.6
6/2	11/83	27.3	18.4	38.0	2.78	84.0
8/5	2/84	35.0	26.3	26.0	7.11	94.4
12/1	5/84	36.9	23.1	31.7	4.77	96.5
13/1	5/84	35.1	23.1	29.4	4.84	92.4
14/1	6/84	30.4	28.4	20.5	4.71	84.0
16/1	7/84	35.7	19.3	20.0	9.78	84.8
17/1	8/84	32.1	22.7	21.9	5.06	81.8
	Means	32.5	23.7	27.0	4.58	87.0
28/1**	11/84	38.6	22.4	17.2	10.62	88.8
32/1**	12/84	—	33.2	19.8	5.81	—

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[*] Average of all analyses performed on various samples of a particular sludge is reported here. Refer to Table A-6 for listing of all analyses.

[†] Ethanol content was very small and varied between 0-6 wt % of the mass tabulated in this column.

[**] Sludges 28/1 and 32/1 were mixtures of Downers Grove primary and Stickney activated sludges.

SECTION 8

STABILITY OF DIGESTER FEEDS

Raw sludge feed slurries stored in a refrigerated feed reservoir were delivered to the digesters by intermittent pumping. The feed reservoir was refrigerated, and the sludge remained at 2° to 4°C to minimize its degradation to acids and gases during the storage period, which varied between 2 and 7 days depending on the HRT of a particular run. The feed reservoir contents were sampled for solids and volatile acids determinations during an 8-day storage period — this storage period is longer than 7 days and represents the worst-case sludge storage. The results of these analyses are reported in Table 37. Looking at the TS and VS analyses, it is clear that these solids concentrations remained essentially unchanged during the 8-day period indicating no significant degradation of the organic materials. The daily samples were analyzed for volatile acids. Concentrations of the individual volatile acids also remained virtually constant except for isobutyric acid, which was present only in low concentrations, during the 8-day storage of the feed slurry. Thus, the information presented in Table 37 showed that the feed sludge composition probably remained quite stable under the conditions of refrigerated storage.

TABLE 37. TIME PROFILES OF SOLIDS AND VOLATILE ACIDS ANALYSES OF DIGESTER FEED SLURRY WHICH WAS PUMPED CONTINUALLY FROM THE REFRIGERATED (4°C) FEED RESERVOIR TO THE ANAEROBIC DIGESTER

Sample type or location	Reservoir level, L	Sample date	Sampler	Day No.	Analysis date	TS, wt %	VS		Volatile acids analysis, mg/L								Ethanol, mg/L
							g/L	wt % of TS	Acetic	Propionic	Isobutyric	Butyric	Isovaleric	Valeric	Caproic	Total as acetic	
Blended batch	--	11/28/83	PP[a]	0	11/25/83	4.5	3.1	68.0	204	220	15	28	53	13	0	450	8
FR[b]	17.89	11/23/83	PP	0	11/25/83	4.4	3.0	68.1	176	222	9	21	14	6	0	388	0
FR	17.89	11/23/83	HPP[c]	0	11/25/83	4.4	3.0	68.3	162	208	13	24	29	5	0	376	0
FR	16.80	11/25/83	HPP	2	11/25/83	4.3	3.1	69.4	179	210	4	17	28	6	0	384	0
FR	14.80	11/26/83	HPP	3	11/26/83	4.3	3.1	70.9	171	206	3	16	13	6	0	363	0
FR	12.80	11/27/83	HPP	4	11/27/83	4.3	3.0	69.5	193	210	12	21	36	7	0	411	0
FR	8.76	11/28/83	HPP	5	11/28/83	4.0	2.8	70.3	196	197	6	17	29	6	0	390	0
FR	5.26	11/29/83	HPP	6	11/30/83	--	--	--	197	190	2	15	22	7	0	380	0
FR	3.51	11/30/83	HPP	7	11/30/83	4.8[d]	3.4	70.6	211	196	3	15	22	7	0	400	0
FR	2.66	12/1/83	HPP	8	12/2/83	4.8[d]	3.3	69.1	246	217	0	17	32	7	0	459	0

[a] PP is peristaltic pump with 1/4-in. I.D. tubing to collect sludge from different areal locations and depths of the reservoir feed slurry.

[b] FR is refrigerated feed reservoir the contents of which were mixed at 86 rpm with a 5-in. diameter 3-blade propeller impellor.

[c] HPP is a hand-operated piston pump which served the same function as that of the peristaltic pump.

[d] The accuracy of these solids analysis was poor because the reservoir liquid levels were very low on these days, and it was difficult to obtain samples of well-mixed feed sludge.

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SECTION 9

THEORETICAL GAS AND METHANE YIELDS OF DIGESTER FEED SLUDGE

Theoretical gas and methane yields were calculated for selected Hanover Park raw feed sludges to estimate the maximum stabilization efficiency corresponding to complete conversion of the organic carbon to methane, carbon dioxide, and microbial cell mass. As discussed below, these calculations can be based on the elemental, COD, or calorific-value analysis of the substrate.

THEORETICAL YIELDS BASED ON ELEMENTAL ANALYSIS

Theoretical total gas and methane yields were calculated for the Hanover Park Lot 1 and Lot 4 mixed activated-primary raw sludges used as digester feeds. These yield calculations were based on the elemental analyses of these sludges and on the following assumptions:

- All of the analyzed total carbon in the feed is biodegradable.
- The feed carbon is incorporated in methane, carbon dioxide, and cellular protoplasm.
- About 30% of the VS is utilized for cell synthesis.
- Complete stoichiometric conversion of the VS is achieved.

As shown in Table 38, the Hanover Park feed sludge had theoretical total gas and methane yields of 0.793 and 0.506 SCM/kg VS reacted (12.7 and 8.1 SCF/lb VS reacted), respectively. The theoretical methane content of the digester gas was calculated to be 64 mol %.

THEORETICAL METHANE YIELDS BASED ON THEORETICAL SLUDGE COD

Theoretical methane yields can be calculated on the basis that each mole of methane produced is tantamount to a removal of two moles of COD ($CH_4 + 2O_2 \rightarrow CO_2 + 2H_2O$). This means that 0.369 SCM (5.91 SCF) of methane produced is equivalent to the removal of 1.0 lb (0.454 kg) of COD. Thus, 369 L of methane production is equivalent to the removal of 1 kg of COD removed. The theoretical COD of the substrate was calculated from the empirical chemical formula of the sludge substrate considering complete chemical oxidation of the measured carbon, nitrogen (TKN), and sulfur, and no oxidation of the phosphorus. This consideration entailed the implicit assumptions that concentrations of sulfate, sulfites, and other oxidized forms can be neglected, and that all the measured phosphorus remains in their oxidized forms; furthermore, it means that a part of the substrate oxygen remains tied

TABLE 38. THEORETICAL GAS AND METHANE YIELDS OF HANOVER PARK SLUDGE BASED ON ELEMENTAL ANALYSES

Sludge lot/batch no.	Elemental analysis, wt % of TS								Theoretical yields, SCM/kg VS reacted		Theoretical methane content, mol %
	Total carbon	Hydrogen	TKN	Total sulfur	Total phosphorus	Oxygen (by diff.)	VS	Ash	Total gas	Methane	
1/1	41.65	6.25	4.99 (0.62)*	1.50	1.46	20.85	73.2	23.30	0.784 [12.56]†	0.493 [7.90]	62.9
4/2	42.87	6.67	4.08	1.24	1.22	19.07	73.9	24.85	0.799 [12.80]	0.517 [8.28]	64.7
								Mean	0.793 [12.7]	0.510 [8.17]	64.3

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* The number in parenthesis is ammonia-nitrogen (NH_3-N); organic nitrogen is (TKN)-(NH_3-N).

† Numbers in brackets are the theoretical yields expressed in units of SCF/lb VS added.

with the phosphorus and is not available for the oxidation of carbon, hydrogen, nitrogen, and sulfur.

The theoretical total and carbonaceous COD's of two raw feed sludges are reported in Table 39. The theoretical total COD is comparable to the analytical COD. The carbonaceous COD, which is less than the total COD, forms the basis for digester methane production calculations. Table 39 indicates that the carbonaceous COD was 88% and 92% of the total COD. On the average, 90% of the total COD was carbonaceous COD.

Theoretical methane yields of raw sludge Lots 1 and 2 were calculated to be 0.506 and 0.487 SCM/kg VS reacted (8.1 and 7.8 SCF/lb VS reacted) corresponding to complete conversion of the carbonaceous substrate COD; these yields compared favorably with those calculated on the basis of elemental analysis. (See Table 37.)

THEORETICAL METHANE YIELDS BASED ON ANALYTICAL COD

Methane yields were also calculated from the measured or analytical COD's of the raw feed sludge samples. These yield calculations are summarized in Table 40.

Based on the theoretical total and the analytical total COD's of raw sludge Lot 1 (2.22 and 1.31 kg COD/kg VS, respectively), it appears that only about 59 wt % of the raw sludge VS was chemically oxidizable. Since biochemical anaerobic oxidation reactions occur under near-ambient temperatures — as opposed to the high temperature of chemical oxidation — it may be reasonable to assume that no more than 59% of the above sludge would be biochemically convertible. Lot 1 raw sludge thus could be about 59% biodegradable.

THEORETICAL METHANE YIELDS BASED ON CALORIFIC VALUE

Theoretical methane yields of the sludge feed could also be calculated from its calorific value (higher heating value) as shown in Table 41. Feed sludge Lots 1 and 4 thus had theoretical methane yields of 0.468 and 0.500 SCM/kg VS reacted (7.49 and 8.01 SCF/lb VS reacted), respectively.

Summary

Methane yields estimated on the basis of elemental analysis, theoretical and analytical COD's, and calorific-value analysis of the feed sludges are summarized in Table 42. Examination of the data in Table 42 shows that theoretical methane yields calculated on the bases of elemental, theoretical COD, and calorific-value analyses of a given feed sludge (e.g., Lots 1 or 4) were within few percentage points of each other. Also, the mean theoretical

TABLE 39. THEORETICAL METHANE YIELDS OF HANOVER PARK SLUDGE BASED ON THEORETICAL CARBONACEOUS COD

Sludge lot no.	VS, % of TS	Empirical chemical formula	Theoretical COD, kg COD/kg VS		Theoretical methane yield*	
			Total	Carbonaceous	SCM/kg VS converted	SCF/lb VS converted
1	73.18	$C_{3.468}H_{6.188}O_{1.301}N_{0.356}S_{0.047}P_{0.047}Ash_x$	2.22	1.95 (87.7)†	0.504	8.07
4	73.9	$C_{3.57}H_{6.604}O_{1.764}N_{0.127}S_{0.089}P_{0.039}Ash_x$	2.04	1.88 (92.2)	0.486	7.78

---

* The theoretical methane yield was calculated from the carbonaceous COD and assuming that 30% of the carbonaceous matter is incorporated in cell mass.

† Numbers in parentheses are the ratios of carbonaceous to total theoretical COD expressed as a percentage.

TABLE 40. THEORETICAL METHANE YIELDS OF HANOVER PARK SLUDGE BASED ON ANALYTICAL CARBONACEOUS COD'S

Feed sludge lot no.	Analytical COD, kg COD/kg VS		Theoretical methane yield,* SCM/kg VS converted (SCF/lb VS converted)
	Total†	Carbonaceous+	
1	1.314	1.183	0.305 (4.89)
5	1.487	1.338	0.345 (5.53)
6	1.580	1.422	0.367 (5.88)
8	1.798	1.618	0.418 (6.69)

* The theoretical methane yields were calculated from the analytical carbonaceous COD's assuming that 30% of the carbonaceous matter is incorporated in cell mass.

† Total COD's in this table are means for the indicated lot.

+ Carbonaceous COD was assumed to be 90% of the total COD.

TABLE 41. THEORETICAL METHANE YIELD OF HANOVER PARK SLUDGE BASED ON CALORIFIC VALUE

Feed sludge lot no.	Mean higher heating value, kcal/kg VS (Btu/lb VS)	Methane yield,* SCM/kg VS reacted (SCF/lb VS reacted)
1	6026 (10,846)	0.468 (7.49)
4	6441 (11,593)	0.500 (8.01)

* The theoretical methane yield was calculated by assuming that 30% of VS is incorporated in the cell mass and that the calorific value of 1 SCM of methane is 9014 kcal (1013 Btu/SCF).

TABLE 42. SUMMARY OF THEORETICAL METHANE YIELDS FOR HANOVER PARK SLUDGE

	Digester feed slurry prepared from lot nos.					
	1	4	5	6	8	Overall mean
Based on elemental analysis						
Carbon dioxide yield,						
SCM/kg VS reacted	0.291	0.282	--	--	--	0.286
SCF/lb VS reacted	4.66	4.51	--	--	--	4.58
						(2.3)[*]
Methane yield,						
SCM/kg VS reacted	0.493	0.518	--	--	--	0.506
SCF/lb VS reacted	7.90	8.29	--	--	--	8.10
						(3.4)
Carbon dioxide content, mol %	36.3	35.2	--	--	--	--
Methane content, mol %	63.7	64.8	--	--	--	--
Methane yield based on theoretical COD,						
SCM/kg VS reacted	0.504	0.486	--	--	--	0.494
SCF/lb VS reacted	8.07	7.78	--	--	--	7.92
						(2.6)
Methane yield based on analyzed COD,						
SCM/kg VS reacted	0.305	--	0.345	0.367	0.418	0.359
SCF/lb VS reacted	4.89	--	5.53	5.88	6.69	5.75
						(13)
Methane yield based on calorific value,						
SCM/kg VS reacted	0.468	0.500	--	--	--	0.484
SCF/lb VS reacted	7.49	8.01	--	--	--	7.75
						(4.7)
Methane methane yield for the lot[†]						
SCM/kg VS reacted	0.488	0.501	--	--	--	0.494
SCF/lb VS reacted	7.82	8.03	--	--	--	7.92
	(3.8)	(3.2)				(1.9)

---

[*] Numbers in parentheses are coefficients of variation.

[†] Methane yields calculated on the bases of elemental analysis, theoretical COD, and calorific value were used in computing the mean.

methane yields of Lots 1 and 4 feed sludges were within about 2.5% of each other. An overall mean of 0.499 SCM/kg VS reacted (7.99 SCF/lb VS reacted) was regarded as the theoretical methane yield for the Hanover Park sludge; the theoretical total gas yield was estimated at 0.787 SCM/kg VS added (12.6 SCF/lb).

The data in Table 42 indicate that theoretical methane yields calculated on the bases of elemental, theoretical COD, and calorific-value analyses were in close agreement with each other. In addition, the theoretical methane yields for the different lots also were about the same. In contrast to these observations, methane yields calculated on the basis of analytical COD's were different for different sludge lots apparently because the oxidizabilities of the lots were different. This observation suggested that the anaerobic biodegradabilities of the various feed sludges were different although they were collected from the same source (Hanover Park sewage treatment plant); the variation in sludge biodegradability may be attributed to the variation in the organic composition of the sludge volatile matter. For example, it can be seen from Table 43 that sludge Lot 1 which had the least analytical-COD based methane yield also had the least ΣCPL, (sum total of the masses of protein, carbohydrate, and lipids). Thus, the higher the ΣCPL, the higher the analytical-COD-based methane yield as indicated by Column 2 of Table 43.

TABLE 43. DEPENDENCE OF POTENTIAL METHANE YIELD OF HANOVER PARK SLUDGE ON PROTEIN, CARBOHYDRATE, AND LIPID CONTENTS

Digester feed slurry prepared from lot no.	Analytical COD-based methane yield, SCM/kg VS reacted (SCF/lb VS reacted)	Theoretical methane yield, SCM/kg VS reacted (SCF/lb VS reacted)	Oxidizability,*	Sum total of protein, carbohydrate and lipids, % of sludge VS
1	0.305 (4.89)	0.494 (7.92)	61.7	81.7
6	0.367 (5.88)	0.494 (7.92)	74.2	84.0
8	0.418 (6.69)	0.494 (7.92)	84.5	94.4

* Oxidizability was the ratio of the analytical COD-based methane yield to the theoretical methane yield.

SECTION 10

BIODEGRADABILITY OF DIGESTER FEED SLUDGE

Anaerobic digestibility potential (ADP) tests were conducted with mesophilic inocula in triplicate with samples of mixed primary-activated feed sludge collected from the Hanover Park wastewater treatment plant of the MSDGC. Details of the test conditions and protocol are described in Section 6. Results of the ADP tests plotted in Figure 14 and shown in Table 44 indicate that after an initial lag, gasification of the Hanover Park Sludge proceeded at a rapid rate for about 17 days after which time there was a dramatic decline in the rate of digestion. This observation seems to indicate that the Hanover Park sludge had one or more highly biodegradable components which were preferentially gasified rapidly during an initial 17-day period. Methane content of the head gases at the end of this vigorous digestion phase was about 73 mol % compared with a methane concentration of 74 mol % at the end of the ADP test. The data in Figure 14 indicate that maximum total gas and methane yields of 0.454 and 0.312 SCM/kg VS added (7.3 and 5.0 SCF/lb VS added) can be expected from digestion of this sludge under mesophilic conditions. A volatile solids reduction of about 48% (which is the higher of the two calculated reductions) was obtained during the ADP digestion test (Table 45). Satisfactory TS, VS, and FS balances were obtained for this ADP test as indicated in Table 45.

As indicated in a previous section, the Hanover Park sludges had a theoretical total gas yield of about 0.787 SCM/kg VS reacted (12.6 SCF/lb VS reacted) representing conversion of all the volatile solids. Comparing this with the long-term ADP total gas yield of about 0.456 SCM/kg VS added (7.3 SCF/lb VS added), it appears that about 58% of the sludge VS was anaerobically biodegradable under mesophilic conditions. This biodegradability factor compares favorably with those reported in the literature. It was concluded in a previous section that about 59% of the sludge carbonaceous matter was chemically oxidizable. This oxidizability factor compared very well with the anaerobic biodegradability of 58%.

As indicated by the ADP test data, a methane yield of about 0.252 SCM/kg VS added (4.1 SCF/lb VS added) was obtained from digestion of the rapidly biodegradable fraction after 35 days of incubation (Figure 14). Comparing this methane yield with the maximum methane yield of 0.312 SCM/kg VS added (5.0 SCF/lb VS added) at the end of the test, it may be reasoned that about 80% of the sludge VS was more rapidly biodegradable than a recalcitrant fraction constituting 20% of the VS.

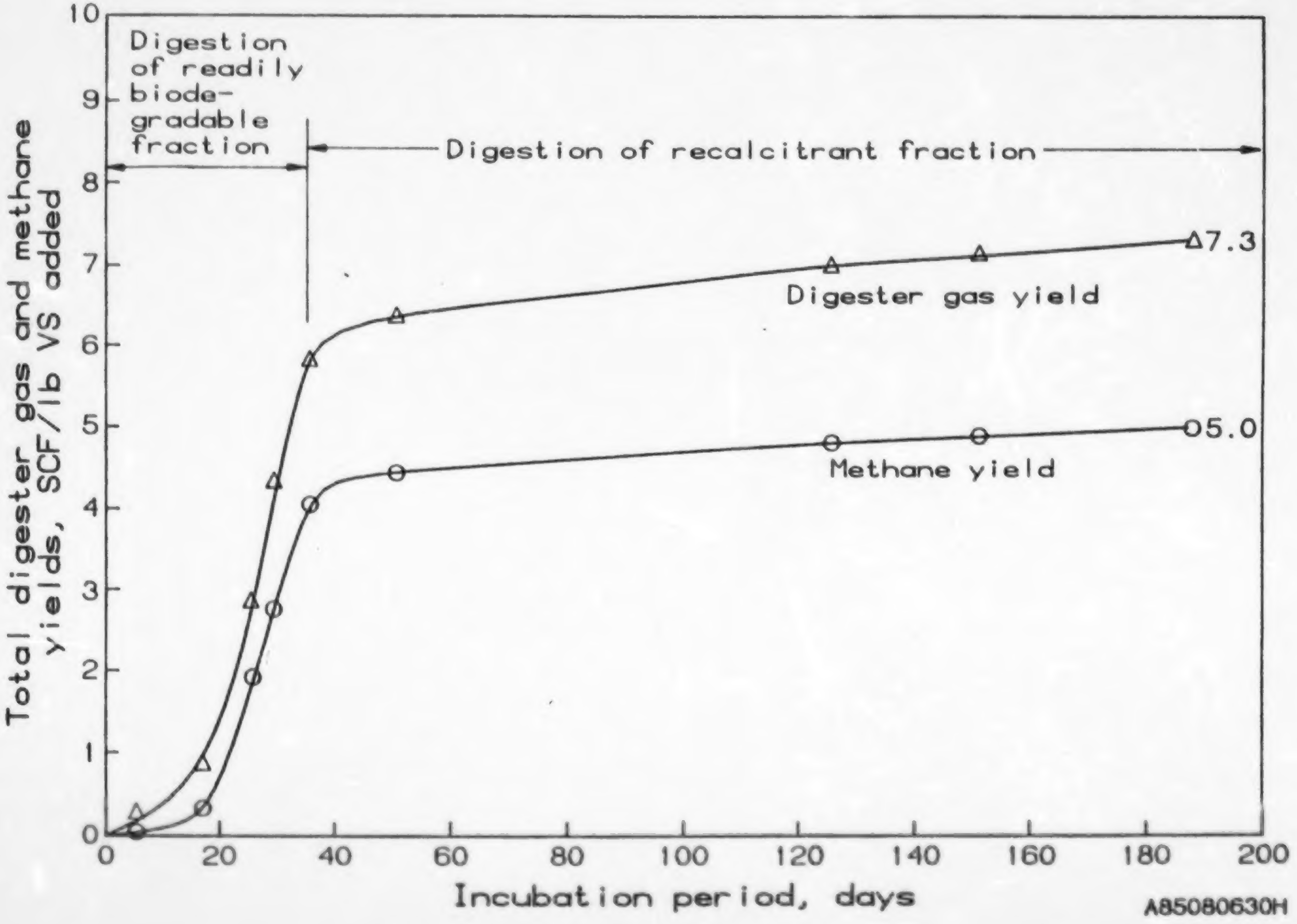

Figure 14. Total digester gas and methane yields from anaerobic digestibility potential (ADP) test conducted at 35°C with Lot 16, Batch 1 Hanover Park mixed activated-primary sludge.

TABLE 44. GAS AND METHANE PRODUCTIONS FROM MESOPHILIC ANAEROBIC DIGESTIBILITY POTENTIAL TEST CONDUCTED WITH LOT 16 BATCH 1 HANOVER PARK SLUDGE*

Incubation period, days	Total gas yield, SCM/kg VS added (SCF/lb VS added)	Methane content, mol %	Methane yield, SCM/kg VS added (SCF/lb VS added)
5.6	0.022 (0.36)	7.9	0.002 (0.03)
17.8	0.052 (0.83)	39.2	0.021 (0.33)
25.8	0.177 (2.83)	68.1	0.120 (1.93)
29.7	0.266 (4.26)	64.4	0.171 (2.74)
34.7	0.360 (5.77)	70.4	0.254 (4.07)
50.0	0.397 (6.36)	69.8	0.277 (4.44)
125.0	0.435 (6.97)	69.0	0.300 (4.81)
150.9	0.444 (7.12)	69.1	0.307 (4.92)
187.9	0.454 (7.27)	68.8	0.312 (5.00)

* Data in this table are the averages of triplicate determinations and are corrected for gas and methane production from the inoculum.

TABLE 45. VOLATILE SOLIDS REDUCTION AND MASS BALANCES FOR THE MESOPHILIC ADP TEST CONDUCTED WITH HANOVER PARK MIXED ACTIVATED-PRIMARY SLUDGE

	Test digesters			Mean
	Replicate 1	Replicate 2	Replicate 3	
Total solids (TS), g				
Initial	0.4451	0.4451	0.4451	0.4451
Final	0.2968	0.2942	0.2780	0.2897
Volatile solids (VS)				
Initial, g	0.3186	0.3186	0.3186	0.3186
% of initial TS	71.57	71.57	71.57	71.57
Final g	0.1756	0.1726	0.1591	0.1691
% of final TS	59.16	58.67	57.23	58.36
VS_R by MOP-16,[*] %	42.5	43.6	46.8	44.3
Mass of digester gas, g				
CO_2	0.0802	0.0882	0.0848	0.0844
Methane	0.0641	0.0694	0.0682	0.0672
Total	0.1443	0.1576	0.1530	0.1516
VS_R by mass of gas,[†] %	45.3	49.5	48.0	47.6
Mass balance,[**] % of initial				
TS	99.1	101.5	96.8	99.2
VS	100.4	103.6	98.0	100.7

---

[*] Volatile solids reduction, VS_R, was calculated by the formula suggested in WPCF Manual of Practice (MOP) 16.[2]

[†] VS_R was assumed to be at least equal to the mass of the dry gas produced.

[**] Mass balance was expressed as the percent ratio of the sum of the masses of material out and product gases to the initial mass of the material.

SECTION 11

PERFORMANCE OF SINGLE-STAGE CFCSTR DIGESTERS

EXPERIMENTAL RUNS

A total of six single-stage high-rate digestion runs were conducted as per the experimental plan to provide baseline data for comparison with two-phase process performance under similar operating conditions. Three mesophilic runs were conducted at mean temperatures between 34.7° and 35.9°C at HRT's of about 15, 7, and 3 days (Table 46). The 7-day- and the 3-day-HRT runs had a higher feed VS concentration than that of the 15-day HRT run because it was felt that substrate concentration should be increased to support higher organism growth rates at the lower HRT's. A second set of three runs was conducted at mean thermophilic temperatures between 54.7° and 56°C and at feed VS concentrations the same as the set of mesophilic runs.

SINGLE-STAGE CFCSTR PROCESS PERFORMANCE

The performance of the mesophilic runs at the three HRT's are compared in Tables 47, 48 and 49. The gas production, effluent quality and digestion-efficiency data in these tables show that the 7-day HRT run was best in terms of organic reduction; this run, although fed with a more concentrated sludge, exhibited a methane yield, gas-phase methane content, alkalinity, and effluent filtrate COD that were comparable with those of the 15-day HRT run.

The mesophilic run conducted at a three-day HRT had lower gas and methane productions and organic reductions, and showed much higher levels of propionic and total VA accumulations indicating unbalanced acidogenic and methanogenic fermentations. Compared to the 7-day HRT test, the 3-day HRT was very unstable and was clearly unsuitable for mesophilic single-stage CFCSTR digestion of sewage sludge.

A comparison of the gas production, effluent-quality, and digestion-efficiency data reported in Tables 50, 51, and 52 for thermophilic single-stage CFCSTR runs showed that the best thermophilic performance was obtained at an HRT of 15 days. Under thermophilic conditions, gas and methane production, and organic reductions decreased and propionate and total VA concentrations increased as the HRT was decreased from 15 to 7 to 3 days. Inhibitory levels of volatile acids were observed for the 3-day HRT test.

The steady-state performances of the mesophilic and thermophilic runs are compared in Table 53, which shows that higher methane yields and production rates were obtained at the thermophilic temperature in all cases. Interestingly, volatile acids and ammonia-nitrogen concentrations of the thermophilic effluents were in all cases higher than those of the mesophilic

TABLE 46. ACTUAL OPERATING CONDITIONS FOR SINGLE-STAGE CFCSTR DIGESTERS FED WITH HANOVER PARK SLUDGE

Run no.	Digester no.	Run duration, days	Steady-state duration, days	Mean culture temp., °C	Mean HRT, days	Mean loading rate, kg VS/m^3-day	Feed total solids conc., g/L	Feed volatile solids conc., g/L
Mesophilic								
SS15M	331	77	57	34.7 (2)†	15.0 (6)	2.00 (14)	40.2	30.1
SS7M	331	90	54	34.9 (2)	7.0 (15)	7.51 (6)	76.1	52.2
SS3M	331	65	19	35.9 (2)	3.1 (8)	15.38 (8)	63.8	48.3
Thermophilic								
SS15T	337	43	25	56.0 (1)	15.0 (3)	2.11 (3)	41.5	31.8
SS7T	331	44	26	55.8 (1)	7.0 (4)	7.10 (4)	68.2	49.9
SS3T	335	25	17	54.7 (1)	3.2 (11)	15.63 (12)	66.7	49.2

* Data reported are the means of all data collected during the steady-state period. All runs were made with Hanover Park sludge but batches were different. See Appendix Table F-1 for specific batch numers.

† Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of the standard deviation to the mean.

TABLE 47. EFFECT OF HRT ON STEADY-STATE GAS PRODUCTIONS FROM MESOPHILIC CFCSTR SINGLE-STAGE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE*

HRT, days	15.0	7.0	3.1
Run No.	SS15M	SS7M	SS3M
Operation			
Feed VS concentration,† mg/L	30,060	52,220	48,290
Loading, kg VS/m^3-day	2.00	7.51	15.38
Performance			
Total gas yield, SCM/kg VS added	0.320 (13)**	0.318 (13)	0.160 (13)
Methane Yield, SCM/kg VS added	0.225 (13)	0.220 (16)	0.089 (15)
Gas Composition, mol %			
Methane	70.3	69.1	55.6
Carbon dioxide	29.2	30.6	43.9
Nitrogen	0.5	0.3	0.5
Total gas production rate, SCM/m^3-day	0.625 (11)	2.327 (12)	2.457 (11)
Methane Production Rate, SCM/m^3-day	0.440 (11)	1.609 (13)	1.365 (13)

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* Data reported are means of all data collected during the steady-state portion of the run.

† Feed VS concentrations are the weighted averages of the various feed slurry concentrations.

** Numbers in parentheses are the coefficients of variation expressed as the percent ratio of the standard deviation to the mean.

TABLE 48. EFFECT OF HRT ON THE QUALITY OF STEADY-STATE EFFLUENTS FROM MESOPHILIC CFCSTR SINGLE-STAGE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE[*]

HRT, days	15.0	7.0	3.1
Run no.	SS15M	SS7M	SS3M
Effluent pH	7.11 (1)[†]	7.06 (0)	6.77 (0)
Alkalinities, mg/L as $CaCO_3$			
Total	6072	6368	6475
Bicarbonate	6071	6196	4620
Volatile acids, mg/L			
Acetic	1	164	343
Propionic	0	104	1571
Isobutyric	0	0	191
Butyric	0	0	39
Isovaleric	0	0	329
Valeric	0	0	86
Caproic	0	0	0
Total as acetic	1 (203)	248 (29)	2017 (20)
Ethanol, mg/L	3	3	0
Nitrogen, mg/L			
Ammonia-N	779	728	1122
Organic-N	1429	1966	2023
Chemical oxygen demand, mg/L			
Total	29,690	67,450	74,791
Filtrate	2024	2956	6331
Solids, mg/L			
TS	35,025	64,350	55,240
VS	22,450	39,585	40,480
TSS	32,710	56,840	—
VSS	23,040	39,460	--
Organic components, mg/L			
Crude protein	8976	10,427	12,644
Carbohydrates	4750	7014	6752
Lipids	5797	11,389	10,397

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[*] Data reported are means of one or more determinations made during the steady-state portion of the run.

[†] Numbers in parentheses are the coefficients of variation expressed as the percent ratio of the standard deviation to the mean.

TABLE 49. EFFECT OF HRT ON STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF MESOPHILIC CFCSTR SINGLE-STAGE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE[a]

HRT, days	15.0	7.0	3.1
Run No.	SS15M	SS7M	SS3M
VS reduction, %			
MOP_{16}[b]	38.2	18.3	13.7
Wt-of-gas basis[c]	28.8	32.7	19.3
VSS reduction, %	26.5	17.2	—
COD (total) reduction, %	31.6	16.3	13.0
Reduction of organic components, %			
Crude protein	27.1	26.2	23.7
Carbohydrates	27.3	26.4	44.4
Lipids	25.1	40.0	16.5
ΣCPL[d]	26.6	32.4	27.6

[a] Data reported are means of all data collected during the steady-state portion of the run. The VSS, COD, and organic component reductions are means of one or more determinations made during the steady-state period.

[b] These VS reductions were calculated according to the following formula: $VS_R' = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[c] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[d] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

TABLE 50. EFFECT OF HRT ON STEADY-STATE GAS PRODUCTIONS FROM THERMOPHILIC CFCSTR SINGLE-STAGE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE[a]

HRT, days	15.0	7.0	3.2
Run No.	SS15T	SS7T	SS3T
HRT, days	15.0	7.0	3.2
Operation			
Feed VS concentration,[b] mg/L	31,760	49,890	49,250
Loading, kg VS/m^3-day	2.11	7.10	15.63
Performance			
Total gas yield,	0.425	0.373	0.180
SCM/kg VS added	(5)[c]	(5)	(9)
Methane Yield,	0.280	0.253	0.114
SCM/kg VS added	(4)	(5)	(9)
Gas composition, mol %			
Methane	66.1	68.0	63.3
Carbon dioxide	33.7	31.9	36.4
Nitrogen	0.2	0.1	0.3
Total gas production rate,	0.894	2.641	2.798
SCM/m^3-day	(4)	(3)	(4)
Methane production rate,	0.591	1.797	1.770
SCM/m^3-day	(4)	(4)	(5)

[a] Data reported are means of one or more determinations made during the steady-state portion of the run.

[b] Feed VS concentrations are the weighted averages of the various feed slurry concentrations.

[c] Numbers in parentheses are the coefficients of variation expressed as the percent ratio of the standard deviation to the mean.

TABLE 51. EFFECT OF HRT ON THE QUALITY OF STEADY-STATE EFFLUENTS FROM THERMOPHILIC CFCSTR SINGLE-STAGE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE[*]

HRT, days	15.0	7.0	3.2
Run no.	SS15T	SS7T	SS3T
Effluent pH	7.47 (1)[†]	7.50) (1)	7.27 (1)
Alkalinities, mg/L as $CaCO_3$			
Total	6500	10,450	9026
Bicarbonate	5854	8683	6440
Volatile acids, mg/L			
Acetic	154	211	1045
Propionic	844	1708	1379
Isobutyric	69	163	375
Butyric	3	0	321
Isovaleric	239	624	811
Valeric	8	0	32
Caproic	9	60	138
Total as acetic	1037 (24)	2105 (13)	3205 (6)
Ethanol, mg/L	0	0	0
Nitrogen, mg/L			
Ammonia-N	1132	1646	1550
Organic-N	868	1460	2212
Solids, mg/L			
TS	27,780	52,320	62,340
VS	18,560	34,310	42,705
Organic components, mg/L			
Crude protein	5425	9125	13,825
Carbohydrates	5444	8625	8428
Lipids	3185	5134	7195

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[*] Data reported are means of one or more determinations made during the steady-state portion of the run.

[†] Numbers in parentheses are the coefficients of variation expressed as the percent ratio of the standard deviation to the mean.

TABLE 52. EFFECT OF HRT ON STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF THERMOPHILIC CFCSTR SINGLE-STAGE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE[a]

HRT, days	15.0	7.0	3.2
Run no.	SS15T	SS7T	SS3T
VS reduction, %			
MOP_{16}[b]	36.8	32.9	19.0
Wt-of-gas basis[c]	45.9	39.6	20.2
Reduction of organic components, %			
Crude protein	53.5	47.8	20.4
Carbohydrate	25.3	10.8	10.4
Lipids	68.2	47.8	22.9
ΣCPL[d]	51.5	38.1	18.4

[a] Data reported are means of all data collected during the steady-state portion of the run. The organic component reductions are means of one or more determinations made during the steady-state period.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 X (VS_i - VS_o)/[VS_i - (VS_i X VS_o)]$.

[c] These VS reductions were calculated according to the following formula: VS_R = 100 X (wt of product gases)/(wt of feed VS).

[d] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

TABLE 53. COMPARISON OF STEADY-STATE PERFORMANCES OF MESOPHILIC AND THERMOPHILIC CFCSTR SINGLE-STAGE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE

HRT, days	15		7		3	
Run no.	SS-15M	SS15-T	SS7M	SS7T	SS3M	SS3T
Culture temperature	Meso	Thermo	Meso	Thermo	Meso	Thermo
Methane yield, SCM/kg VS added	0.225	0.280	0.220	0.253	0.089	0.114
Methane Production Rate, SCM/m^3-day	0.440	0.591	1.609	1.797	1.365	1.770
Methane content, mol %	70.3	66.1	69.1	68.0	55.6	63.3
Effluent volatile acids, mg/L as acetic	1	1037	248	2105	2017	3205
Effluent pH	7.11	7.47	7.06	7.50	6.77	7.27
VS reduction, %						
MOP_{16}[a]	38.2	36.8	18.3	32.9	13.7	19.0
wt-of-gas basis[b]	28.8	45.9	32.7	39.6	19.3	20.2
Carbon-in-gas basis[c]	29.1	39.4	28.8	34.7	14.9	16.7
Based on theoretical gas yield[d]	29.7	39.4	29.5	24.6	14.8	16.7
Biodegradable VS reduction[e]	51.2	68.0	50.9	59.7	25.6	28.8
Organic reductions, %						
Crude protein	27.1	53.5	26.2	47.8	23.7	20.4
Carbohydrates	27.3	25.3	26.4	10.8	44.4	10.4
Lipids	25.1	68.2	40.0	47.8	16.5	22.9
ΣCPL[f]	26.6	51.5	32.4	38.1	27.6	18.4

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[a] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (VS_i - VS_o)/[VS_R - (VS_i \times VS_o)]$.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[c] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (1.84 X wt of carbon in product gas)/(wt of VS fed).

[d] These VS reductions are calculated by expressing the observed total gas yield as a percentage of the theoretical gas yield of 1.078 SCM/kg VS added.

[e] The biodegradable VS reduction was calculated by dividing the theoretical gas yield based VS reduction by a biodegradability fraction of 0.58.

[f] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

effluents suggesting a higher degree of solids liquefaction at the higher temperature. Despite the prevalence of higher volatile acids concentrations in the thermophilic digesters, the culture pH remained significantly above 7 apparently because the thermophilic process generated higher buffering capacity as evidenced by the higher concentration of ammonium bicarbonate.

Overall, thermophilic digestion exhibited higher gas yields and production rates and stabilization efficiencies than those of mesophilic digestion. However, it is questionable whether this increased performance is sufficient to justify process operation at a higher temperature. To justify thermophilic operation it may be necessary to operate the digester at much higher feed solid concentrations and to develop means of reducing the effluent VA.

Carbohydrate reduction was highest at the lowest HRT during mesophilic digestion; the reverse was true for thermophilic digestion. In general, carbohydrate reduction at the thermophilic temperature was about the same or lower than that at the mesophilic temperature. Crude protein reductions at the thermophilic and mesophilic temperatures were comparable at the lowest HRT; higher crude protein reductions were obtained at the thermophilic temperature at an HRT's of 15 and 7 days. At the mesophilic temperature, the highest lipid conversion was obtained at an HRT of 7 days. Lipid reductions at the thermophilic temperature were higher than those at the mesophilic temperature at all HRT's.

COMPARISON OF CPL CONVERSIONS UNDER MESOPHILIC CONDITIONS

The percent conversions of carbohydrates, proteins, and lipids (CPL) were about the same as each other at a 15-day HRT. By comparison, O'Rourke[72] observed lower "effective" destruction of influent "degradable" protein than of influent "degradable" lipid.

Protein conversion was the same as the carbohydrate conversion at the 7-day HRT. Also, the conversion of the organic components at the 15-day and the 7-day HRT's were about the same. Lipid conversion at the 7-day HRT was higher than those of protein and carbohydrate. Lipids degradation was lowest at the low HRT; a similar observation was made by O'Rourke.[72] Surprisingly, carbohydrate degradation was maximum at the lowest HRT of 3 days.

COMPARISON OF CPL CONVERSION UNDER THERMOPHILIC CONDITIONS

Under thermophilic conditions protein degradation was maximized at the 7-day HRT. At the other two HRT's protein reductions were about the same as those under mesophilic conditions. Lipid reduction was maximum at the 15-day HRT and decreased with decreases in the HRT as opposed to being maximized at the 7-day HRT.

Overall, the combined conversions of carbohydrate protein and lipid at the thermophilic temperature were higher than those at the mesophilic temperature at the 15-day and the 7-day HRT; the reverse was true at the 3-day HRT. Since the thermophilic methane yields were higher than mesophilic yields, it is apparent that the overall rate and yield of digestion (i.e., hydrolysis, acidification, and gasification) is higher at thermophilic temperature than at mesophilic temperature. However, the thermophilic cultures were unable to convert all of the incoming volatile acids to gas considering the significantly higher effluent volatile acids concentrations observed at the thermophilic temperature.

MASS BALANCES

Volatile solids mass balances (defined as the ratio of sum of the masses of effluent VS and gases to influent VS) performed with the steady-state data showed that the effluent VS's were between 100% and 110% of influent VS. Fixed solid balances (defined as the ratio of effluent FS to influent FS) showed that effluent FS's were between 93% and 109% of the influent FS. Details of the mass balances are shown in Figures B-1 to B-6.

SECTION 12

PERFORMANCE OF CFCSTR TWO-PHASE DIGESTION SYSTEMS

EXPERIMENTAL RUNS

Three sets of two-phase digestion runs (designated as meso-meso, meso-thermo, and thermo-thermo in Table 54) were conducted with CFCSTR acid- and methane-phase digesters at HRT's of about 15, 7, and 3 days. A meso-meso digestion run was conducted with a mesophilic acid digester operated in series with a mesophilic methane digester. Similarly, a meso-thermo system consisted of a mesophilic acid-phase digester followed by a thermophilic methane digester and so on. The acid-phase digesters had an HRT of about 2 days except when the system HRT was 3 days. For two-phase system HRT's of 3 days, the acid-phase digester had an HRT of about 0.9 days. As with the single-stage CFCSTR digestion runs (Section 11), higher feed VS concentrations were used for the 7-day and the 3-day HRT runs.

The run durations and the steady-state durations for all experiments were, in general, longer than three HRT's. The variabilities of the operating parameters were generally lower than 15% for the digester runs listed in Table 54.

PERFORMANCE OF MESO-MESO SYSTEMS

Acid-Phase Performance

The performance of the meso-meso two-phase systems at 15-, 7- and 3-day HRT's are compared in Tables 55, 56, and 57 in terms of gas production and quality, effluent quality, and organic-reduction efficiency. The acid digester generally exhibited the highest gas and methane yields when it was operated at a 2-day HRT and was charged with the higher VS concentration (47 and 50 g/L) feeds; gas and methane yields were lower at the lower HRT (0.8-0.9 days) or with the lower feed VS concentration. Residual volatile acids concentration in the acid digester effluent was much higher at the 0.9-day HRT than at an HRT of 2 days indicating that acidogenesis was favored over gasification at lower HRT's. Gas and methane production rates from the acid-phase digesters increased in direct proportion to the increase in loading rates. Methane production was observed in all acid-phase runs, although the methane content of the head gases decreased as the HRT decreased or the feed VS concentration increased. It appears that methane production from the acid-phase digester was predominantly by the hydrogen-oxidizing methane bacteria which, by virtue of their higher growth rate, could survive at the low HRT's. Methane formation by the aceticlastic reaction is not expected to be significant at HRT's of 2 days or lower.

TABLE 54. ACTUAL STEADY-STATE OPERATING CONDITIONS FOR PROCESS COMPARISON CFCSTR TWO-PHASE DIGESTION SYSTEMS OPERATED WITH HANOVER PARK SLUDGE[*]

Run no.	Digester nos.	Total run duration, days	Steady-state run duration, days	Mean acid digester temp., °C	Mean methane digester temp., °C	Mean methane acid-digester HRT, days	Mean methane-digester HRT, days	Mean system HRT, days	Mean system loading, kg VS/m^3-day	Feed total solids concentration, g/L	Feed volatile solids concentration, g/L
Meso-Meso											
TP15M-M	332-333	70	45	34.9 (3)[†]	34.8 (2)	2.0 (14)	13.2 (15)	15.2 (15)	1.94 (14)	41.8	29.5
TP7M-M	334-333	52	23	35.3 (3)[†]	35.7 (1)	1.9 (12)	4.9 (12)	6.8 (12)	7.29 (13)	67.7	49.7
TP3M-M[**]	334-333	26	13	35.7 (2)	35.0 (1)	0.91 (8)	2.15 (10)	3.06 (9)	14.7 (9)	66.4	44.9
Meso-Thermo											
TP15M-T	334-337	70	38	35.1 (2)	55.7 (1)	2.1 (10)	13.0 (3)	15.1 (3)	2.14 (5)	45.8	32.3
TP7M-T	334-331	52	24	35.4 (2)	55.4 (2)	1.9 (17)	5.5 (17)	7.4 (18)	6.74 (17)	66.4	50.0
Thermo-Thermo											
TP7T-T	335-331	50	27	54.4 (1)	54.8 (1)	2.1 (18)	6.0 (18)	8.1 (18)	6.80 (19)	69.9	51.3
TP3T-T	335-331	19	9	55.3 (1)	52.9 (3)	0.81 (13)	2.33 (13)	3.14 (13)	15.0 (14)	68.0	47.2

[*] Data reported are the means of all data collected during the steady-state period. Specific batch numbers for all runs are given in Appendix Table F-1.

[†] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of the standard deviation to the mean.

[**] This run was conducted with mixed Downers Grove primary and Stickney activated sludges.

TABLE 55. EFFECT OF HRT ON STEADY-STATE GAS PRODUCTIONS FROM MESO-MESO CFCSTR TWO-PHASE DIGESTION OF HANOVER PARK SLUDGE[a]

	Two-phase at 15.2-day HRT			Two-phase at 6.8-day HRT			Two-phase at 3.1-day HRT[b]		
	Acid digester	Methane digester	System	Acid digester	Methane digester	System	Acid digester	Methane digester	System
Run no.	TP15M-M			TP7M-M			TP3M-M		
Operation									
Feed VS concentration,[c] mg/L	29,500	--	--	49,680	--	--	46,600	--	--
HRT, days	2.0	13.2	15.2	1.9	4.9	6.8	0.91	2.15	3.06
Loading, kg VS/m^3-day	14.78	2.23	1.94	26.38	10.08	7.29	51.21	21.68	15.23
Performance									
Total gas yield, SCM/kg VS added	0.093 (18)[d]	0.499 (16)	0.592 (16)	0.157 (16)	0.310 (11)	0.467 (12)	0.108 (7)	0.197 (9)	0.305 (6)
Methane yield, SCM/kg VS added	0.058 (19)	0.352 (16)	0.410 (16)	0.091 (16)	0.212 (12)	0.302 (13)	0.057 (10)	0.124 (9)	0.180 (8)
Gas composition, mol %									
Hydrogen	--	--	--	0.0	--	--	0.05	0.0	0.0
Methane	62.6	70.6	69.3	57.1	68.2	64.7	52.0	63.1	59.1
Carbon dioxide	36.1	28.9	30.1	42.5	31.6	35.1	47.3	36.5	40.4
Nitrogen	1.3	0.5	0.6	0.4	0.2	0.2	0.7	0.4	0.5
Total gas production rate, SCM/m^3-day	1.345 (12)	1.090 (9)	1.124 (8)	4.063 (5)	3.089 (5)	3.358 (4)	5.575 (6)	4.293 (13)	4.674 (12)
Methane production rate, SCM/m^3-day	0.841 (12)	0.770 (9)	0.779 (8)	2.343 (5)	2.100 (5)	2.173 (4)	2.898 (9)	2.708 (14)	2.764 (12)

[a] The data reported are means of all data collected during the steady-state portion of the run.

[b] This run was conducted with mixed Downers Grove primary and Stickney activated sludge.

[c] Feed VS concentrations are the weighted averages of the various feed slurry concentrations.

[d] Numbers in parentheses are the coefficients of variation expressed as the percent ratio of the standard deviation to the mean.

TABLE 56. EFFECT OF HRT ON THE QUALITY OF STEADY-STATE EFFLUENTS FROM MESO-MESO CFCSTR TWO-PHASE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE[a]

	Two-phase at 15.2-day HRT		Two-phase at 6.8-day HRT		Two-phase at 3.1-day HRT[b]	
	Acid digester	Methane digester[c]	Acid digester	Methane digester[c]	Acid digester	Methane digester[c]
Run no.	TP15M-M		TP7M-M		TP3M-M	
HRT, days	2.0	13.2	1.9	4.9	0.91	2.15
Effluent pH	6.63	7.10	6.63	7.30	6.48	7.19
	(1)[d]	(1)	(1)	(1)	(1)	(1)
Alkalinities, mg/L as $CaCO_3$						
Total	3290	4950	7475	8100	6550	8350
Bicarbonate	2926	4944	6368	8068	2059	6735
Volatile acids, mg/L						
Acetic	295	20	721	63	2177	218
Propionic	328	8	728	29	1403	1503
Isobutyric	40	0	109	0	288	50
Butyric	24	0	160	0	749	0
Isovaleric	84	0	136	0	596	195
Valeric	15	0	61	10	1907	136
Caproic	0	0	31	33	51	31
Total as acetic	663	26	1627	109	5518	1680
	(33)	(112)	(15)	(44)	(16)	(15)
Ethanol, mg/L	3	4	0	0	19	0
Nitrogen, mg/L						
Ammonia-N	621	646	918	1049	1138	1820
Organic-N	970	795	2241	1845	1927	1682
Chemical oxygen demand, mg/L						
Total	35,760	26,080	--	--	--	--
Filtrate	2270	958	--	--	--	--
Solids, mg/L						
TS	37,050	30,355	60,720	50,690	61,600	57,450
VS	25,520	18,535	42,480	32,810	39,320	35,040
TSS	30,900	28,120	--	--	--	--
VSS	23,960	19,890	--	--	--	--
Organic components, mg/L						
Crude protein	6094	4183	14,006	11,531	12,044	10,512
Carbohydrates	5375	4404	7791	4932	9224	5413
Lipids	3458	1705	7178	3824	6448	5879

[a] Data reported are means of one or more determinations made during the steady- state period.

[b] This run was conducted with mixed Downers Grove primary and Stickney activated sludges.

[c] System effluent characteristics are the same as those of the methane-phase.

[d] Numbers in parentheses are the coefficients of variation expressed as the percent ratio of the standard deviation to the mean.

TABLE 57. EFFECT OF HRT ON STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF MESO-MESO CFCSTR TWO-PHASE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE[a]

	Two-phase at 15.2-day HRT			Two-phase at 6.8-day HRT			Two-phase at 3.1-day HRT[b]		
	Acid digester	Methane digester	System	Acid digester	Methane digester	System	Acid digester	Methane digester	System
Run no.	TP15M-M			TP7M-M			TP3M-M		
HRT, days	2.0	13.2	15.2	1.9	4.9	6.8	0.91	2.15	3.06
VS reduction, %									
MOP$_{16}$[c]	11.4	29.2	37.2	15.7	21.2	33.6	17.1	11.4	26.5
Wt-of-gas basis[d]	10.7	58.0	63.4	18.1	33.4	51.5	13.5	26.1	35.5
VSS reduction, %	11.6	17.0	26.6	--	--	--	--	--	--
COD (total) reduction, %	9.6	27.1	34.1	--	--	--	--	--	--
Reduction of organic components, %									
Crude protein	31.4	31.4	52.9	9.6	17.7	25.6	33.0	12.7	41.5
Carbohydrate	35.3	18.1	47.0	29.1	36.6	55.1	15.8	41.3	50.6
Lipids	49.2	50.7	75.0	28.2	46.7	61.8	19.7	8.8	26.8
ΣCPL[e]	37.8	31.0	57.1	20.6	30.0	44.4	25.0	21.3	41.0

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[a] The data reported are means of one or more determinations made during the steady-state period.

[b] This run was operated with mixed Downers Grove primary and Stickney activated sludges.

[c] These VS reduction were calculated according to the following formula: $VS_R = 100 X (VS_i - VS_o)/[VS_i X VS_o)]$.

[d] These VS reduction were calculated according to the following formula: VS_R = 100 X (wt of product gases)/(wt of VS fed).

[e] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

The acid-digester gases did not contain any hydrogen except during operation at an HRT of 0.9 days, indicating that the rate of hydrogen utilization by the syntrophic methane formers exceeded the rate of hydrogen production by oxidation of the particulate sludge substrates. In all cases, the acid-phase head gases contained more nitrogen than the methane digester gases, indicating that reduction of nitrogen oxides to nitrogen gas (denitrification) was favored in the acid digester. This is expected because substrate oxidation in the acid-phase digester is expected to be coupled with hydrogen removal by denitrifiers.

The pH's of the acid-phase digesters stabilized at about 6.5-6.6. The bicarbonate alkalinity was maximum (about 6400 mg/L as $CaCO_3$) at a 2-day HRT and with 50 g/L VS content feed, and it decreased as the flow-through rate increased or the feed VS concentration decreased.

Carbohydrate and lipid reductions were higher at the higher HRT of 2 days, whereas crude protein reduction appeared to be lower at the 2-day HRT.

Methane-Phase Performance

Total gas and methane yields from the methane digesters decreased as the methane-phase HRT was decreased from 13 to 5 to 2 days; conversely, the gas and methane production rates increased with decreases in HRT and increases in the loading rate.

It is noteworthy that methane production from the methane digester amounted to 86% of the system methane production when the ratio of methane-digester HRT to system-HRT was 0.87; similarly, 70% and 69% of system methane production emanated from the methane digester when the ratios of methane-digester HRT to two-phase system HRT were 0.72 and 0.69, respectively. Thus, about 70% or more of the system methane production emanated from the methane phase of the meso-meso two-phase process.

The methane content of the methane-digester gases were significantly higher than those of the acid digesters. The pH and alkalinity of the methane digesters were considerably higher than those of the acid digesters. The methane digesters received a low bicarbonate alkalinity-content acidic feed. The bicarbonate alkalinity increased substantially during methane fermentation, which had the effect of scrubbing CO_2 and was responsible, in part, for enhancing the methane content of the methane digester gas.

It is interesting to note that ΣCPL (sum of the masses of carbohydrate, protein and lipid) reductions in the methane digesters were lower than those in the acid digester indicating that liquefaction was more predominant in the acid digester. The ΣCPL reductions at the 7-day and the 3-day HRT's were about the same during two-phase operation.

Performance of the Overall System

Total gas and methane yield from the meso-meso two-phase system decreased as the loading rate was increased from 1.9 to 15.2 kg VS/m^3-day and the corresponding reduction in HRT from 15 to 3 days. Gas and methane production rates at 7- and 3-day HRT's increased substantially by 390% and 270%, respectively, compared to that observed at a 15-day HRT. The overall protein, carbohydrate, and lipid reduction (ΣCPL) decreased progressively as the two-phase system HRT was decreased. However, the decrease in ΣCPL reduction was not significant when the HRT was decreased from 7 days to 3 days.

PERFORMANCE OF MESO-THERMO SYSTEMS

Acid-Phase Performance

Two meso-thermo two-phase runs were conducted at system HRT's of about 15 and 7 days (Tables 58 and 59). Gas and methane yields and the gas-phase methane contents of the acid-phase digester decreased and residual volatile acid concentration increased as the feed VS concentration was increased from about 32 to 50 mg/L. Thus, acid formation was enhanced by the more concentrated feed. The gas and methane production rates from the acid-phase digester increased almost in direct proportion to the increase in the loading rate. The acid digester gases had high methane contents; no hydrogen was detected in the gas phase. The two acid-phase runs at an HRT of about 2 days exhibited a pH of about 6.6 and high bicarbonate alkalinity and ammonia nitrogen concentrations (Table 59). Carbohydrate and lipid reductions increased while protein reduction decreased almost in direct proportion to the increase in feed VS concentration (Table 60).

Methane-Phase Performance

Gas and methane yields and production rates as well as effluent volatile acids concentrations were higher at a 5.5-day HRT than at a 13-day HRT, while organic reductions at these two HRT's were nearly equal. Methane production from the thermophilic methane digester amounted to 67% of the system methane production when the ratio of the methane digester HRT to two-phase system HRT was 0.86. About 72% of the system methane production emanated from this methane digester when the methane digester-HRT to system-HRT ratio was 0.74. Overall, thermophilic methane digester operation at a 5.5-day HRT with a concentrated feed appeared to be more attractive than that at a 13-day HRT.

Total carbohydrate-protein-lipid (ΣCPL) reductions in the methane digester were higher than those in the acid digesters; this indicated continued and enhanced liquefaction-acidification under the thermophilic conditions of the methane digester. By comparison, the meso-meso two-phase system showed that ΣCPL reduction in the acid-phase digester was much higher than that in the methane-phase digester, and that little liquefaction occurred in the methane digester.

Performance of the Overall System

Gas and methane yields and production rate from the 7-day HRT meso-thermo two-phase process were higher than those at a 15-day HRT, while ΣCPL reductions at these two HRT's were about the same; volatile solids reduction was higher at the lower HRT. Overall, better meso-thermo two-phase performance was observed at a 7-day system HRT.

TABLE 58. EFFECT OF HRT ON GAS PRODUCTIONS FROM STEADY-STATE MESO-THERMO TWO-PHASE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE[a]

	Two-phase at 15.1-day HRT			Two-phase at 7.4-day HRT		
	Acid digester	Methane digester	System	Acid digester	Methane digester	System
Run no.	-----------	TP15M-T	----------	-----------	TP7M-T	-----------
Operation						
Feed Concentration,[b] mg/L	32,300	--	--	49,965	--	--
HRT, days	2.1	13.0	15.1	1.9	5.5	7.4
Loading, kg VS/m^3-day	15.52	2.48	2.14	26.29	9.06	6.74
Performance						
Total gas yield, SCM/kg VS added	0.163	0.290	0.453	0.156	0.342	0.498
	(11)[c]	(10)	(7)	(17)	(12)	(13)
Methane Yield, SCM/kg VS added	0.104	0.198	0.302	0.087	0.231	0.318
	(10)	(10)	(7)	(17)	(13)	(13)
Gas Composition, mol %						
Hydrogen	0.0	0.0	0.0	0.0	0.0	0.0
Methane	63.7	68.3	66.8	56.0	67.4	63.8
Carbon dioxide	35.9	31.3	32.8	43.7	32.3	35.9
Nitrogen	0.4	0.4	0.4	0.3	0.3	0.3
Total gas production rate, SCM/m^3-day	2.514	0.720	0.954	3.997	2.993	3.251
	(5)	(10)	(6)	(7)	(10)	(7)
Methane Production Rate, SCM/m^3-day	1.604	0.492	0.637	2.235	2.022	2.077
	(6)	(10)	(6)	(7)	(10)	(7)

[a] Data reported are the means of all data collected during the steady-state portion of the run.

[b] Feed VS concentrations are the weighted averages of the various feed slurries used during the steady-state portion of the run.

[c] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of standard deviation to the mean.

TABLE 59. EFFECT OF HRT ON STEADY-STATE EFFLUENT QUALITIES OF MESO-THERMO CFCSTR TWO-PHASE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE[a]

	Two-phase at 15.1-day HRT		Two-phase at 7.4-day HRT	
	Acid digester	Methane digester[b]	Acid digester	Methane digester[b]
Run no.	----- TP15M-T	------	------ TP7M-T	------
HRT, days	2.1	13.0	1.9	5.5
Effluent pH	6.64	7.54	6.66	7.57
	(2)[c]	(1)	(1)	(1)
Alkalinities, mg/L as $CaCO_3$				
Total	4952	7092	6050	7975
Bicarbonate	4094	6443	4651	6246
Volatile acids, mg/L				
Acetic	497	179	892	441
Propionic	408	672	849	1415
Isobutyric	28	32	27	94
Butyric	62	0	159	0
Isovaleric	81	185	147	361
Valeric	22	0	27	0
Caproic	33	26	32	69
Total as acetic	966	867	1826	1900
	(17)	(13)	(13)	(18)
Ethanol, mg/L	0	0	0	0
Nitrogen, mg/L				
Ammonia-N	756	1249	874	1452
Organic-N	1642	1094	2365	1706
Solids, mg/L				
TS	39,590	31,710	60,550	49,330
VS	27,240	20,020	42,760	31,950
Organic components, mg/L				
Crude protein	10,262	6838	14,781	10,662
Carbohydrates	4601	4228	7487	6700
Lipids	6306	2264	9875	3568

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[a] Data reported are means of one or more determinations made during the steady-state period.

[b] System effluent characteristics are the same as those of the methane-phase.

[c] Numbers in parentheses are the coefficients of variation expressed as the percent ratio of the standard deviation to the mean.

TABLE 60. EFFECT OF HRT ON STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF MESO-THERMO CFCSTR TWO-PHASE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE[a]

	Two-phase at 15.1-day HRT			Two-phase at 7.4-day HRT		
	Acid digester	Methane digester	System	Acid digester	Methane digester	System
Run no.	----------	TP15M-T	----------	----------	TP7M-T	----------
HRT, days	2.1	13.0	15.1	1.9	5.5	7.4
VS reduction, %						
MOP_{16}[b]	7.3	22.4	28.0	12.1	23.5	32.8
Wt-of-gas basis[c]	18.1	36.2	48.5	18.9	40.8	54.7
Reduction of organic components, %						
Crude protein	12.8	33.4	41.9	8.3	27.9	33.8
Carbohydrates	24.5	8.1	30.6	34.0	10.5	40.9
Lipids	7.6	64.1	66.8	12.7	63.9	68.5
ΣCPL[d]	14.2	37.0	46.0	17.1	35.0	46.0

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[a] The data reported here are means of one or more determinations made during the steady-state period.

[b] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[c] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[d] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

PERFORMANCE OF THERMO-THERMO SYSTEMS

Acid-Phase Performance

As with the mesophilic acid digesters, gas and methane yields from the thermophilic acid digester decreased and gas and methane production rates increased as the HRT was decreased from 2.1 to 0.81 days (Table 61). The acid digester gases had methane contents of 57-58 mol %, and no hydrogen was detected even at an HRT of 0.81 days. As with the meso-meso system, the acid digester gases contained much higher nitrogen contents than those of the methane digesters. This observation indicated that the hydrogen removal rate of the thermophilic syntrophic methane formers was higher than the substrate oxidation-linked hydrogen production rate even at a low HRT of 0.8 days. In contrast, there was evidence of hydrogen accumulation during mesophilic acid-phase digestion at a comparable HRT of 0.9 days. This observation suggests that thermophilic syntrophic methane formers have a higher growth rate than those of the mesophilic syntrophic methanogens. The pH and volatile acid concentrations in the thermophilic acid digesters were higher than those in the mesophilic acid digesters under similar operating conditions. (See Tables 56, 59, and 62.) Acetate was detected at the highest concentration followed by propionic and other higher acids. These results provided evidence of enhanced liquefaction-acidification under thermophilic conditions.

Methane-Phase Performance

Gas and methane yields from the thermophilic methane digester decreased by 18-20% when the HRT was reduced from 6 days to 2.33 days, and even at these low HRT's the digester gases had a methane content of 68%. A very high gas production rate of 4.1 vol/culture vol-day was observed at an HRT of 2.33 days. About 81-84% of the thermo-thermo system methane production was obtained from the thermophilic methane digester for methane digester-HRT to two-phase system-HRT ratios of 0.43-0.74. By comparison, a lower proportion of the system methane production was derived from the mesophilic methane digester of the meso-meso two-phase process under similar HRT conditions. This difference was due to depressed methane production in the thermophilic acid digester relative to that in the mesophilic acid digester.

COMPARISON OF MESO-MESO, MESO-THERMO, AND THERMO-THERMO TWO-PHASE SYSTEMS

Systems Comparison at a 15-Day HRT

A compilation of the summary data collected at a system HRT of 15 days shows that the meso-meso two-phase system was better than the meso-thermo system with respect to gas and methane yields and production rates, gas-phase methane content, and VS and ΣCPL reductions (Table 63). The meso-thermo two-phase process had a much higher effluent volatile acid concentration than that of the meso-meso two-phase process and yet the effluent pH of the former system was higher than that of the latter; this is explained by the fact that the ammonia-nitrogen concentration and the bicarbonate alkalinity of the meso-thermo process were considerably higher than those of the meso-meso two-phase process. About 86% of the system methane production from the meso-meso two-phase process was derived from the methane digester compared with 67% for the

TABLE 61. EFFECT OF HRT ON STEADY-STATE GAS PRODUCTIONS FROM CFCSTR THERMO-THERMO TWO-PHASE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE[a]

	Two-phase at 8.1-day HRT			Two-phase at 3.1-day HRT		
	Acid digester	Methane digester	System	Acid digester	Methane digester	System
Run no.	TP7T-T			TP3T-T		
Operation						
Feed concentration,[b] mg/L	51,340	—	—	47,240	—	—
HRT, days	2.1	6.0	8.1	0.81	2.33	3.14
Loading, kg VS/m^3-day	33.90	8.53	6.80	58.41	20.24	15.03
Performance						
Total gas yield, SCM/kg VS added	0.071 (20)[c]	0.253 (16)	0.324 (16)	0.048 (30)	0.207 (15)	0.255 (11)
Methane yield, SCM/kg VS added	0.042 (22)	0.177 (16)	0.219 (16)	0.027 (28)	0.141 (16)	0.168 (11)
Gas composition, mol %						
Hydrogen	—	—	—	0.0	0.0	0.0
Methane	58.1	70.2	67.6	57.2	68.2	65.9
Carbon dioxide	40.9	29.4	31.9	41.2	31.7	33.8
Nitrogen	1.0	0.4	0.5	1.6	0.1	0.3
Total gas production rate, SCM/m^3-day	1.715 (14)	2.119 (16)	2.015 (12)	2.814 (30)	4.133 (12)	3.793 (8)
Methane production rate, SCM/m^3-day	0.993 (13)	1.485 (16)	1.358 (12)	1.606 (28)	2.818 (12)	2.506 (8)

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[a] Data reported are the means of all data collected during the steady-state portion of the run.

[b] Feed VS concentrations are the weighted averages of the various feed slurries used during the steady-state portion of the run.

[c] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of standard deviation to the mean.

TABLE 62. EFFECT OF HRT ON STEADY-STATE EFFLUENT QUALITIES OF THERMO-THERMO CFCSTR TWO-PHASE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE[a]

	Two-phase at 8.1-day HRT		Two-phase at 3.1-day HRT	
	Acid digester	Methane digester[b]	Acid digester	Methane digester[b]
Run no.	----- TP7T-T ------		----- TP3T-T ------	
HRT, days	2.1	6.0	0.81	2.33
Effluent, pH	6.88 (1)[c]	7.59 (1)	6.92 (1)	7.59 (1)
Volatile acids, mg/L				
Acetic	994	474	1192	102
Propionic	723	878	667	910
Isobutyric	215	209	197	40
Butyric	198	0	268	0
Isovaleric	465	359	446	190
Valeric	5	34	164	233
Caproic	31	41	48	59
Total as acetic	2154 (24)	1580 (12)	2432 (31)	1146 (29)
Ethanol, mg/L	0	0	0	5
Wt-of-gas VS reduction,[d] %	8.1	25.5 [33.7][e]	5.6	21.5 [27.1]

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[a] Data reported are means of one or more determinations made during the steady-state period.

[b] System effluent characteristics are the same as those of the methane-phase.

[c] Numbers in parentheses are the coefficients of variation expressed as the percent ratio of the standard deviation to the mean.

[d] The wt-of-gas VS reduction was calculated according to the following formulas: VS_R = 100 X (wt of product gases)/(wt of VS feed).

[e] Numbers in brackets are VS reductions for the system.

TABLE 63. COMPARISON OF STEADY-STATE PERFORMANCES OF MESO-MESO AND MESO-THERMO CFCSTR TWO-PHASE DIGESTION SYSTEMS OPERATED AT A 15-DAY HRT WITH HANOVER PARK SLUDGE

Culture temperatures	Meso-meso	Meso-thermo
Run no.	TP15M-M	TP15M-T
Methane yield, SCM/kg VS added	0.410	0.302
Methane production rate, SCM/m^3-day	0.779	0.637
Methane content, mol %	69.3	66.8
Methane production from methane digester, % of system methane production	86.0	67.2
Effluent volatile acids, mg/L as acetic	26	867
Ammonia-N, mg/L	646	1249
Effluent pH	7.10	7.54
Methane digester bicarbonate alkalinity, mg/L as $CaCO_3$	4944	6443
VS reduction, %		
MOP$_{16}$[a]	37.2	28.0
Wt-of-gas basis[b]	63.4	48.5
Carbon-in-gas basis[c]	53.8	40.9
Based on theoretical yield[d]	54.9	42.0
Biodegradable VS reduction[e]	94.7	72.4
Organic reductions, %		
Crude protein	52.9	41.9
Carbohydrates	47.0	30.6
Lipids	75.0	66.8
ΣCPL[f]	57.1	46.0

[a] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[c] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (1.84 \times$ wt of carbon in product gas)/(wt of VS fed).

[d] These VS reductions are calculated by expressing the observed total gas yield as a percentage of the theoretical gas yield of 1.078 SCM/kg VS added.

[e] The biodegradable VS reduction was calculated by dividing the theoretical gas yield based VS reduction by a biodegradability fraction of 0.58.

[f] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

thermophilic digester of the meso-thermo system. Overall, the above observations indicated that the bulk of the two-phase system methane production was derived from the methane digester at both mesophilic and thermophilic temperatures. The volatile acids and ammonia nitrogen concentrations and crude protein and lipid productions in the thermophilic methane digester were higher than those of the mesophilic methane digester, and yet the thermophilic digester exhibited lower methane production. These observations indicated that the thermophilic methane digester enhanced particulate hydrolysis and acidification, but the hydrolysis products were probably not as efficiently gasified as it was by the mesophilic methane digester. To achieve enhanced thermophilic process performance, it may be necessary to develop special thermophilic culture acclimation techniques, and to apply novel digester designs to provide microbial SRT's that are considerably higher than the HRT. Since thermophilic organisms have higher growth rates than mesophiles, a higher-concentration feed should be used for the thermophilic system.

Systems Comparison at a 7-Day HRT

Comparing meso-meso, meso-thermo, and thermo-thermo two-phase operation at a system HRT of 7 days, the meso-meso system was best in terms of methane production rate and residual volatile acid concentration (Table 64). The meso-thermo two-phase process exhibited slightly higher VS, protein, lipid,and ΣCPL reductions and had higher effluent volatile acids and ammonia nitrogen concentrations than those of the meso-meso two-phase process although the methane yields from both systems were the same. Similar observations were made during operation at a 15-day HRT. It may be inferred from these results that there was a higher degree of liquefaction-acidification under thermophilic conditions, and that the products of these reactions could not be gasified by the thermophilic methanogens as efficiently as it was by their mesophilic counterparts. From the above trends and characteristics of thermophilic operation it would be expected that a thermo-thermo two-phase process exhibited the lowest methane yield and production rate when compared with the meso-meso and the meso-thermo systems.

As was the case with the 15-day HRT runs, the thermophilic methane digesters had higher effluent acids and also a higher pH than those of the mesophilic methane digester. This may be explained by the fact that the ammonia nitrogen concentration at the thermophilic temperature was higher than that at the mesophilic temperature.

As reported in Table 64, 70-80% of the two-phase system methane production was derived from the methane digester at both mesophilic and thermophilic temperatures.

Systems Comparison at a 3-Day HRT

The performances of the meso-meso and thermo-thermo two-phase systems at an HRT of about 3 days are compared in Table 65. The data show the same trends as observed at the higher HRT's in that the meso-meso two-phase process exhibited higher methane yield and production rate than those of the thermo-thermo system. As with other system HRT's discussed above, 69-84% of the two-phase methane production was derived from the methane digester.

TABLE 64. COMPARISON OF STEADY-STATE PERFORMANCES OF CFCSTR MESO-MESO, MESO-THERMO, AND THERMO-THERMO TWO-PHASE DIGESTION SYSTEMS OPERATED AT A 7-DAY HRT WITH HANOVER PARK SLUDGE

Culture temperatures	Meso-meso	Meso-thermo	Thermo-thermo
Run no.	TP7M-M	TP7M-T	TP7T-T
Methane yield, SCM/kg VS added	0.302	0.318	0.219
Methane production rate, SCM/m^3-day	2.173	2.077	1.358
Methane content, mol %	64.7	63.8	67.6
Methane production from methane digester, % of system methane production	69.7	72.3	81.2
Effluent volatile acids, mg/L as acetic	109	1900	1580
Ammonia-N, mg/L	1049	1452	--
Effluent pH	7.30	7.57	7.59
Methane digester bicarbonate alkalinity, mg/L as $CaCO_3$	8068	6246	--
VS reduction, %			
MOP$_{16}$[a]	33.6	32.8	--
Wt-of-gas basis[b]	51.5	54.7	33.6
Carbon-in gas basis[c]	42.8	45.1	29.6
Based on theoretical yield[d]	43.3	46.2	30.1
Biodegradable Vs reduction[e]	74.7	79.6	51.8
Organic reductions, %			
Crude protein	25.6	33.8	--
Carbohydrates	55.1	40.9	--
Lipids	61.8	68.5	--
ΣCPL[f]	44.4	46.0	--

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[a] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (wt of product gas)/(wt of VS fed).

[c] These VS reductions were calculated according to the following formula: $VS_R = 110 \times (1.84 \times$ wt of carbon in product gas)/(wt of VS fed).

[d] These VS reductions are calculated by expressing the observed total gas yield as a percentage of the theoretical gas yield of 1.078 SCM/kg VS added.

[e] The biodegradable VS reduction was calculated by dividing the theoretical gas yield based VS reduction by a biodegradability fraction of 0.58.

[f] ΣCPL means the sum of the masses of carbohydrate, crude protein, and lipids.

TABLE 65. COMPARISON OF STEADY-STATE SYSTEM PERFORMANCES OF MESO-MESO AND THERMO-THERMO CFCSTR TWO-PHASE DIGESTION SYSTEMS OPERATED AT A 3-DAY HRT WITH CHICAGO SLUDGE

Culture temperatures	Meso-meso	Thermo-thermo
Run no.	TP3M-M	TP3T-T
Feed sludge[a]	DG-S	HP
Methane yield, SCM/kg VS added	0.180	0.168
Methane production rate, SCM/m^3-day	2.764	2.506
Methane content, mol %	59.1	65.9
Methane production from methane digester, % of system methane production	68.8	83.5
Effluent volatile acids, mg/L as acetic	1680	1146
Effluent pH	7.19	7.59
VS reduction, %		
MOP_{16}[b]	26.5	--
Wt-of-gas basis[c]	35.5	27.1
Carbon-in-gas basis[d]	28.5	23.5
Based on theoretical yield[e]	28.3	23.6
Biodegradable VS reduction[f]	48.8	40.8
Organic reductions, %		
Crude protein	41.5	--
Carbohydrates	50.6	--
Lipids	26.8	--
ΣCPL[g]	41.0	--

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[a] DG-S means mixed Downers Grove primary and Stickney activated sludges; HP means Hanover Park sludge.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[c] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[d] These VS reductions were calculated according to the following formulas: $VS_R = 100 \times (1.84 \times$ wt of carbon in product gas)/(wt of VS fed).

[e] These VS reductions are calculated by expressing the observed total gas yield as a percentage of the theoretical gs yield of 1.078 SCM/kg VS added.

[f] The biodegradable VS reduction was calculated by dividing the theoretical gas yield based VS reduction by a biodegradability fraction of 0.58.

[g] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

Summary

Overall, the results of the various two-phase experiments showed that meso-meso two-phase operation seemed to be better than meso-thermo or thermo-thermo two-phase operation in terms of gas production. The meso-thermo and the thermo-thermo two-phase processes afforded higher organic reduction, but lower gas yields than those of the meso-meso process. Gasification reactions appeared to be less efficient under thermophilic conditions.

The acid digesters performed well at the 2-day and the 0.8-day HRT's at both temperatures; however, acid accumulation was higher at the thermophilic temperature while gas production was lower compared to those at the mesophilic temperature.

The mesophilic methane digester exhibited higher gas yields than the thermophilic methane digester at all HRT's. Although there was evidence of enhanced liquefaction-acidification at the thermophilic temperature, gasification of these reaction products was inefficient in a CFCSTR digester. For overall system operation, HRT's of 1-2 days for the acid-phase and about 5-6 days for the methane-phase seemed optimum.

SECTION 13

PROCESS COMPARISON: CFCSTR SINGLE-STAGE VERSUS CFCSTR TWO-PHASE

PROCESS COMPARISON AT A 15-DAY HRT

The performances of the single-stage CFCSTR and the two-phase CFCSTR runs at a 15-day HRT are compared in Table 66. Both the meso-thermo and meso-meso two-phase processes exhibited better performances than mesophilic or thermophilic single-stage conventional high-rate digestion in terms of total gas and methane yields and production rates, VS reduction, and conversion of the carbohydrate fraction of VS (Figure 15). The methane yields of the meso-meso and meso-thermo two-phase processes were 82% and 34% higher than those of the mesophilic single-stage run (15-day HRT baseline run). Similarly, the methane production rates from the meso-meso and meso-thermo two-phase processes were about 77% and 47% higher than those of the single-stage processes.

Volatile solids reductions for the single-stage and overall two-phase systems were calculated by four different methods as explained in Table 66. It appears that VS reductions calculated by the MOP-16 method were unrealistic and inconsistent with the gas production data. This is probably due to the lack of accuracy of the VS measurement procedure, particularly when alkali and acids were added for pH control and/or sample preservation. The MOP-16 VS reductions tended to be lower than those calculated from mass balances on theoretical gas yields. Volatile solids reductions calculated on the bases of carbon content of digester gas and theoretical gas yield were within 2% of each other, and appeared to be more realistic than those obtained by the MOP-16 formula. Biodegradable VS reduction were calculated to afford a more realistic assessment of process performance. These calculations were based on an estimated biodegradability factor of 0.58 (see Section 10).

Information presented in Table 66 and Figure 15 shows that both meso-meso and meso-thermo two-phase systems exhibited higher VS reductions than the single-stage systems, and that the meso-meso two-phase system had the highest VS and organics reductions. Considering conversions of individual organic components, proteins and lipids were degraded at higher efficiencies than carbohydrates under all conditions except in the case of the 15-day HRT single-stage mesophilic run where all organic components were degraded about equally.

The meso-meso two-phase digestion run exhibited the best performance in all respects relative to the other runs. The observed methane yield from this run was 82% of the theoretical compared with 60%, 56% and 45% for the meso-thermo two-phase, single-stage mesophilic, and single-stage thermophilic runs,

TABLE 66. COMPARISON OF STEADY-STATE PERFORMANCES OF CFCSTR SINGLE-STAGE AND TWO-PHASE DIGESTION SYSTEMS OPERATED AT ABOUT A 15-DAY SYSTEM HRT WITH HANOVER PARK SLUDGE

	Single-stage mesophilic	Single-stage thermophilic	Meso-thermo two-phase	Meso-meso two-phase
Run no.	SS15M	SS15T	TP15M-T	TP15M-M
Total gas yield, SCM/kg VS added	0.320	0.425	0.453	0.592
Methane yield, SCM/kg VS added	0.225	0.280 (24.4)[a]	0.302 (34.2)	0.410 (82.2)
Observed methane yield as:				
% of theoretical yield	45.0	56.0	60.4	82.0
% of ADPT yield	70.8	88.1	95.0	128.9
Methane production rate, SCM/m^3-day	0.440	0.591 (34.3)	0.637 (46.8)	0.779 (77.0)
Methane content, mol %	70.3	66.1	66.8	69.3
Effluent volatile acids, mg/L as acetic	1	1037	867	26
Effluent pH	7.11	7.47	7.54	7.10
Ammonia-nitrogen, mg/L	779	1132	1249	646
Ratio of effluent bicarbonate alkalinity to feed bicarbonate alkalinity	1.53	2.71	2.75	1.73
VS reduction, %				
MOP_{16}[b]	38.2	36.8	28.0	37.2
Wt-of-gas basis[c]	28.8	45.9	48.5	63.4
Carbon-in-gas basis[d]	29.1	39.4	40.9	53.8
Based on theoretical gas yield[e]	29.7	39.4	42.0	54.9
Biodegradable VS reduction[f]	51.2	68.0	72.4	94.7
Organic reduction, %				
Crude protein	27.1	53.5	41.9	52.9
Carbohydrates	27.3	25.3	30.6	47.0
Lipids	25.1	68.2	66.8	75.0
ΣCPL[g]	26.6	51.5	46.0	57.1

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[a] Numbers in parentheses are the percentage increases in the particular performance parameter over that of the mesophilic single-stage high-rate process.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[c] These VS reductions were calculated according to the following formula: VS_R = 100 X (wt of product gases)/(wt of VS fed).

[d] These VS reductions were calculated according to the following formula: VS_R = 100 X (1.84 X wt of carbon in product gas)/(wt of VS fed).

[e] These VS reductions are calculated by expressing the observed total gas yield as a percentage of the theoretical gas yield of 1.078 SCM/kg VS added.

[f] The biodegradable VS reduction was calculated by dividing the theoretical gas-yield based VS reduction by a biodegradability factor of 0.58.

[g] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

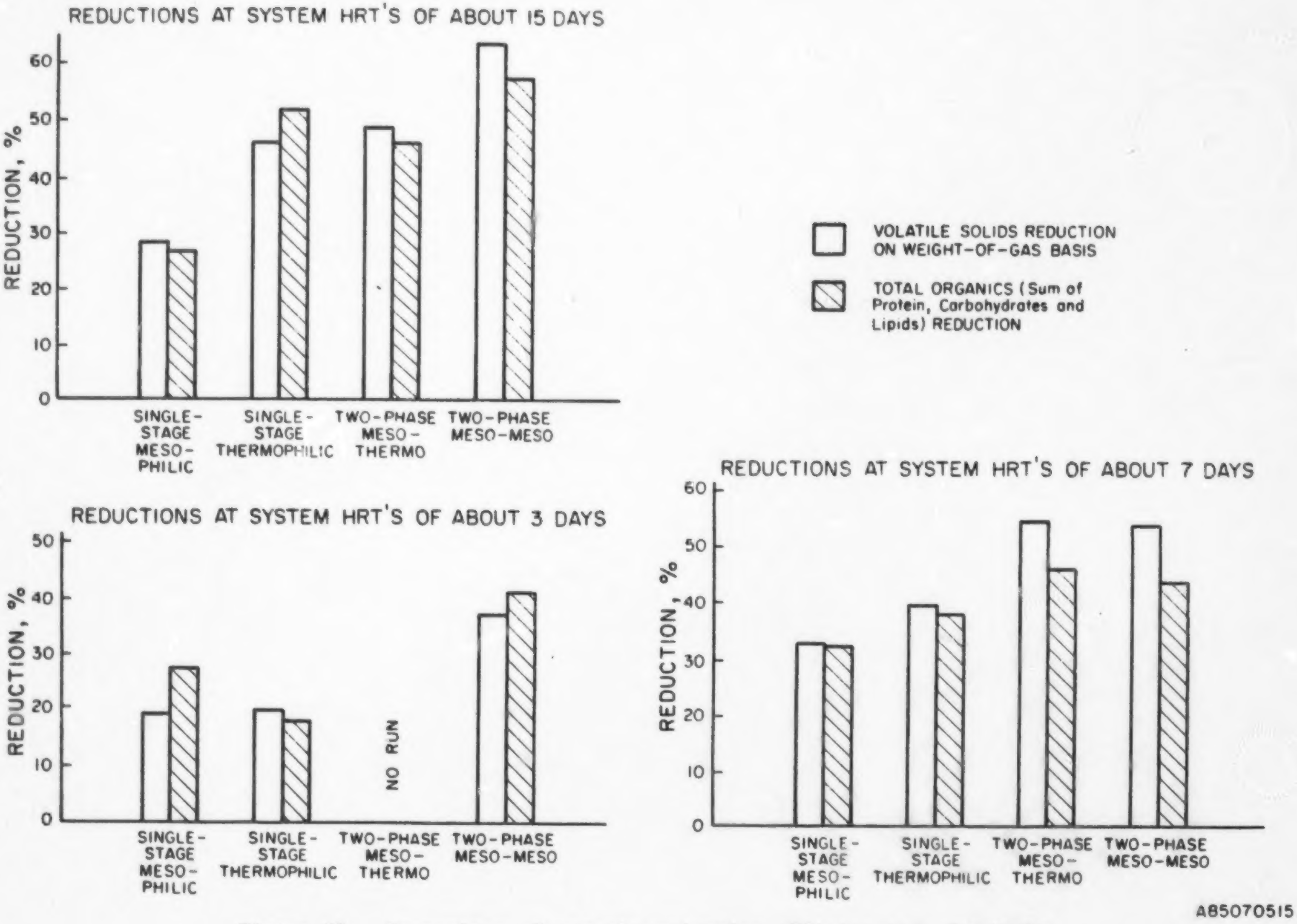

Figure 15. Comparison of organic reduction efficiencies of CFCSTR single-stage and two-phase anaerobic digestion systems.

respectively. The meso-meso two-phase process effected complete conversion of all the biodegradable volatile solids.

PROCESS COMPARISON AT A 7-DAY HRT

Table 67 compares the performances of mesophilic and thermophilic single-stage digestion with those of thermo-thermo, meso-thermo, and meso-meso two-phase runs, all at a 7-day system HRT. The summary data show that the meso-thermo two-phase system exhibited the best performance in terms of gas yield and VS and organic reductions. The next best performance at a 7-day HRT was exhibited by the meso-meso two-phase process. Surprisingly, the thermo-thermo two-phase process was worse than the meso-thermo and the meso-meso two-phase runs. Comparison of the methane yields of methane digesters of the three two-phase systems in Table 67 shows that the gasification efficiencies of the methane digesters were comparable when they were operated in tandem with mesophilic acid-phase digesters; but the methane digester gas production declined sharply when it was preceded by a thermophilic acid-phase digester. A plausible explanation for this phenomenon is that thermophilic acid-phase digestion products retarded acetate and/or CO_2-H_2 conversion directly; alternatively, acetogenic conversion of higher fatty acids and other acetate precursors to acetate could have been inhibited.

Lipid and carbohydrate degradations were higher for two-phase digestion than for single-stage digestion. Crude protein and lipid conversions were enhanced under thermophilic conditions. Carbohydrate reduction seemed to be higher at the mesophilic temperature. The efficiency of lipid conversion was higher than those of protein and carbohydrate.

Overall, the performances of the single-stage and two-phase systems at the 15- and 7-day HRT's were essentially similar with the exception that the meso-meso two-phase process performed significantly better at the 15-day HRT.

PROCESS COMPARISON AT A 3-DAY HRT

In contrast to system operation at HRT's of 15 and 7 days, the meso-meso two-phase run exhibited the best performance at a 3-day system HRT (Table 68), although it should be noted that a meso-thermo run was not conducted at this HRT. Both the meso-meso and thermo-thermo two-phase processes were better than the mesophilic and thermophilic single-stage runs at this low HRT. While protein and lipid degradations were favored at a thermophilic temperature, carbohydrate conversion was higher than those of protein and lipids at the mesophilic temperature.

It is noteworthy that at a 3-day HRT the methane yield of the meso-meso two-phase process was 102% higher than that of the single-stage process; this increase was higher than those observed at system HRT's of 7 and 15 days. Thus, the relative advantages of two-phase digestion were more conspicuous at the shortest HRT.

TABLE 67. COMPARISON OF STEADY-STATE PERFORMANCES OF CFCSTR SINGLE-STAGE AND TWO-PHASE DIGESTION SYSTEMS OPERATED AT ABOUT A 7-DAY HRT WITH HANOVER PARK SLUDGE

	Single-stage mesophilic	Single-stage thermophilic	Thermo-thermo two-phase	Meso-thermo two-phase	Meso-Meso two-phase
Run no.	SS7M	SS7T	TP7T-T	TP7M-T	TP7M-M
Total gas yield, SCM/kg VS added	0.318	0.373	0.324	0.498	0.467
Methane yield, SCM/kg VS added	0.220	0.253 (15.0)[a]	0.219 (0.0)	0.318 (44.6)	0.302 (37.3)
Observed methane yield as:					
% of theoretical yield	44.0	50.6	43.8	63.6	60.4
% of ADPT yield	69.2	79.5	68.9	100.0	95.0
Methane production rate, SCM/m^3-day	1.609	1.797 (11.7)	1.358 (-15.6)	2.077 (29.1)	2.173 (35.0)
Methane content, mol %	69.1	68.0	67.6	63.8	64.7
Effluent volatile acids, mg/L as acetic	248	2105	1580	1900	109
Effluent pH	7.06	7.50	7.59	7.57	7.30
Ammonia-nitrogen, mg/L	728	1646	--	1452	1049
Ratio of effluent bicarbonate alkalinity to feed bicarbonate alkalinity	1.43	2.26	--	1.41	2.09
VS feduction, %					
HOP_{16}[b]	13.3	32.9	--	32.8	33.6
Wt-of-gas basis[c]	32.7	39.6	33.6	54.7	51.5
Carbon-in-gas basis[d]	28.8	34.7	29.6	45.1	42.8
Based on theoretical methane yield[e]	29.5	34.6	30.1	46.2	43.3
Biodegradable VS reduction[f]	50.9	59.7	51.8	79.6	74.7
Organic reduction, %					
Crude protein	26.2	47.8	--	33.8	25.6
Carbohydrates	26.4	10.8	--	40.9	55.1
Lipids	40.0	47.8	--	68.5	61.8
ΣCPL[g]	32.4	38.1	--	46.0	44.4

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[a] Numbers in parenthesis are the percentage increases in the particular performance parameter over that of the mesophilic single-state high-rate process.

[b] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[c] These VS reductions were calculated according to the following formula:
VS_R = 100 X (wt of product gases)/(wt of VS fed).

[d] These VS reductions were calculated according to the following formula:
VS_R = 100 X (1.84 X wt of carbon in product gas)/(wt of VS fed).

[e] These VS reductions are calculated by expressing the observed total gas yield as a precentage of the theoretical gas yield of 1.078 SCM/kg VS added.

[f] The biodegradable VS reduction was calculated by dividing the theoretical gas yield based VS reduction by a biodegradability fraction of 0.58.

[g] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

TABLE 68. COMPARISON OF STEADY-STATE PERFORMANCES OF CFCSTR SINGLE-STAGE AND TWO-PHASE DIGESTION SYSTEMS OPERATED AT ABOUT A 3-DAY HRT WITH HANOVER PARK SLUDGE

	Single-stage mesophilic	Single-stage thermophilic	Thermo-thermo two-phase	Meso-meso two-phase[a]
Run no.	SS3M	SS3T	TP3T-T	TP3M-M
Total gas yield, SCM/kg VS added	0.160	0.180	0.255	0.305
Methane yield, SCM/kg VS added	0.089	0.116 (28.1)[b]	0.168 (88.8)	0.180 (111.2)
Observed methane yield as:				
% of theoretical yield	17.8	22.8	33.6	36.0
% of ADFT Yield	28.0	35.8	52.8	56.6
Methane production rate, SCM/m^3-day	1.365	1.770 (29.7)	2.506 (83.2)	2.764 (102.5)
Methane content, mol %	55.6	63.3	65.9	59.1
Effluent volatile acids, mg/L as acetic	2017	3205	1146	1680
Effluent pH	6.77	7.27	7.59	7.19
Ammonia-nitrogen, mg/L	1122	1550	--	1820
Ratio of effluent bicarbonate alkalinity to feed bicarbonate alkalinity	1.88	2.35	--	5.41
VS reduction, %				
MOP_{16}[c]	13.7	19.0	--	26.5
Wt-of-gas basis[d]	19.3	20.2	27.1	35.5
Carbon-in-gas basis[e]	14.9	16.7	23.5	26.5
Based on theoretical gas yield[f]	14.8	16.7	23.6	28.3
Biodegradable VS reduction[g]	25.6	28.8	40.8	48.8
Organic reduction, %				
Crude protein	23.7	20.4	--	41.5
Carbohydrates	44.4	10.4	--	50.6
Lipids	16.5	22.9	--	26.8
ΣCPL[h]	27.6	18.4	--	41.0

[a] This run was operated with mixed Downers Grove primary and Stickney activated sludges.

[b] Numbers in parenthesis are the percentage increases in the particular performance parameter over that of the mesophilic single-stage high-rate process.

[c] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[d] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[e] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times (1.84 \times$ wt of carbon in product gas)/(wt of VS fed).

[f] These VS reductions were calculated by expressing the observed total gas ield as a percentage of the theoretical gas yield of 1.078 SCM/kg VS added.

[g] The biodegradable VS reduction was calculated by dividing the theoretical gas-yield-based VS reduction by a biodegradability factor of 0.58.

[h] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

CHARACTERISTICS OF THERMOPHILIC DIGESTION

Examination of the data in Tables 66, 67, and 68 shows that essentially all thermophilic systems exhibited much higher residual effluent volatile acids than the mesophilic systems. However, acids accumulations under thermophilic conditions were higher in the single-stage runs at HRT's of 7 and 3 days than in the two-phase runs. These observations suggested the following:

- Volatile acids were not gasified as efficiently under thermophilic conditions as they were at the mesophilic temperature.

- Under thermophilic conditions, the efficiency of volatile acids conversion by two-phase digestion seemed to be higher than that by single-stage digestion.

It was reasoned that acids accumulation under thermophilic conditions probably occurred due to one or both of the following reasons:

- Inhibition of acetogenic bacteria and retarded conversion of higher fatty acids and other acetate-precursors to acetate

- Inhibition of acetate-utilizing methane bacteria and retarded conversion of acetate to methane and carbon dioxide.

The effluent volatile acids data shown in Table 69 indicated that under similar operating conditions, the thermophilic acid digester experienced higher accumulation of acetate, butyrate, and iso-valerate than the mesophilic acid-phase digester, suggesting that utilization of these acids was retarded under thermophilic fermentation conditions. It is probable that acetate-utilizing methanogens, and butyrate and valerate-utilizing acetogens were inhibited more during thermophilic digestion than they were during mesophilic digestion.

The phenomenon of acids accumulation was clearly evident in the thermophilic methane digesters of the two-phase systems (Table 70). Whereas there was hardly any volatile acids accumulation in the mesophilic methane digester at HRT's of 13 and 5 days, acetate, propionate, iso-butyrate, iso-valerate, and caproate accumulated at much higher concentrations during thermophilic metabolism. Interestingly, butyrate which tended to accumulate in the thermophilic acid-digester did not accumulate at all in the methane digester. Also, there was no accumulation of valeric acid in the thermophilic methane digester. Thus, butyrate and valerate were metabolized at the higher HRT's prevalent in the methane digester. Under thermophilic conditions propionate accumulated in the highest concentration followed by acetate (Table 70). It is noteworthy that accumulation of all the acids increased almost in direct proportion to the increase in dilution rate as the HRT was decreased from 13 days to 5 days. The above observations indicated the following effects of thermophilic digestion:

- Aceticlastic gasification (acetate conversion) was retarded during acid-phase digestion at a short (2-day) HRT and during methane-phase digestion

TABLE 69. STEADY-STATE EFFLUENT VOLATILE ACIDS CONCENTRATIONS IN MESOPHILIC AND THERMOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 2-DAY HRT

	Mesophilic acid-phase		Thermophilic acid-phase
Two-phase system run no.	TP7M-M	TP7M-T	TP7T-T
Feed VS concentration, g/L	49.7	50.0	51.3
Volatile acids, mg/L			
Acetic	721	892	994
Propionic	728	849	723
Isobutyric	109	27	215
Butyric	160	159	198
Isovaleric	136	147	465
Valeric	61	27	5
Caproic	31	32	31
Total as acetic	1627	1826	2154

at even higher HRT's of 5 and 13 days suggesting that thermophilic methanogens are inhibited by certain metabolite(s) of thermophilic digestion

- Propionate-utilizing acetogens were inhibited even at a high HRT of 13 days

- Butyrate and valerate metabolism was not affected under thermophilic conditions, but caproate conversion was affected even at a high HRT of 13 days

- Thermophilic bacteria which utilize the branch-chain fatty acids (isobutyrate and iso-valerate) experienced inhibition even at high HRT's

- The above inhibitory effects at the thermophilic temperature of 55°C increased in direct proportion to the increase in dilution rate.

It is indeed true that acetate-utilizing methanogens and propionate-, caproate-, iso-butyrate-, and iso-valerate-utilizing acetogens are inhibited in the methane-phase digesters under thermophilic conditions, then higher accumulations of these very acids would also be predicted for thermophilic single-stage digesters. Data presented in Table 71 show that the experimental observations were in total agreement with these predictions. As expected from the above considerations, acids accumulations in the single-stage thermophilic digester were considerably higher than those in the single-stage mesophilic

TABLE 70. STEADY-STATE EFFLUENT VOLATILE ACIDS CONCENTRATIONS IN MESOPHILIC AND THERMOPHILIC CFCSTR METHANE-PHASE DIGESTERS OF TWO-PHASE SYSTEMS FED WITH HANOVER PARK SLUDGE*

	13-day HRT		5-day HRT	
	Mesophilic methane-phase	Thermophilic methane-phase	Mesophilic methane-phase	Thermophilic methane-phase
Two-phase system run no.	TP15M-M	TP15M-T	TP7M-M	TP7M-T
System feed VS concentration, g/L	29.5	32.3	49.7	50.0
Volatile acids, mg/L				
Acetic	20	179	63	441
Propionic	8	672	29	1415
Isobutyric	0	32	0	94
Butyric	0	0	0	0
Isovaleric	0	185	0	361
Valeric	0	0	10	0
Caproic	0	26	33	69
Total as acetic	26	867	109	1900

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* The acid-phase digesters of the two-phase systems were operated at 2-day HRT's.

TABLE 71. STEADY-STATE VOLATILE ACIDS CONCENTRATIONS IN MESOPHILIC AND THERMOPHILIC SINGLE-STAGE CFCSTR DIGESTERS OPERATED WITH HANOVER PARK SLUDGE

	15-day HRT		7-day HRT		3-day HRT	
	Mesophilic single-stage	Thermophilic single-stage	Mesophilic single-stage	Thermophilic single-stage	Mesophilic single-stage	Thermophilic single-stage
Run no.	SS15M	SS15T	SS7M	SS7T	SS3M	SS3T
Feed VS concentration, g/L	30.1	31.8	52.2	49.9	48.3	49.2
Volatile acids, mg/L						
Acetic	1	154	164	211	343	1045
Propionic	0	844	104	1708	1571	1379
Isobutyric	0	69	0	163	191	375
Butyric	0	3	0	0	39	321
Isovaleric	0	239	0	624	329	811
Valeric	0	8	0	0	86	32
Caproic	0	9	0	60	0	138
Total as acetic	1	1037	248	2105	2017	3205
Methane yield, SCM/kg VS added	0.225	0.280	0.220	0.253	0.089	0.114
VS reduction, %	28.8	45.9	32.7	39.6	19.3	20.2
SCPL reduction, %	26.6	51.5	32.4	38.1	27.6	18.4

digester, although methane yield, VS reduction, and total carbohydrate-protein-lipid (SCPL) reduction under thermophilic conditions were significantly higher than those of mesophilic digestion. By comparison, acid accumulations in the thermophilic two-phase systems were significantly lower than those in the single-stage process under comparable conditions of HRT and feed VS concentration.

In light of the above discussions, it may be concluded that relative to mesophilic conditions, the thermophilic temperature enhanced the hydrolysis of the particulate matter and acidification of the hydrolysate, but retards acetogenic conversion of propionate, branch-chain acids, and caproate as well as acetate fermentation with the result that these metabolites accumulated in the digester. This problem was aggravated more as the HRT of the thermophilic digester is reduced. Volatile acids accumulation in thermophilic two-phase anaerobic digestion systems were significantly lower than those in the single-stage thermophilic process. This indicated that the thermophilic two-phase process promoted enhanced acetogenic and methanogenic activities than conventional single-stage digestion.

The acid-phase digesters of the two-phase systems were operated at HRT's of about 1.9-2.1 and 0.8-0.9 days which were expected to be lower than the critical HRT for acetate-utilizing methanogens. Consequently, the methanogenic activity in the acid-phase digesters would be primarily attributable to that of the hydrogen-utilizing methanogens. A concern that is worth considering is whether the hydrogen-utilizing methane bacteria were also adversely affected by thermophilic metabolites in a similar manner as the acetogens and acetate-utilizing methanogens were impacted during thermophilic digestion. Table 72 was prepared to elucidate this question. Considering thermophilic and mesophilic acid-phase digestion under similar operating conditions, there was evidence of a higher degree of acidification at the thermophilic temperature. Yet, methane yield at the thermophilic temperature was less than one-half that at the mesophilic temperature. It may be inferred from these observations that certain thermophilic sludge-degradation products could also be inhibitory to the hydrogen-oxidizing methanogens. Alternatively, the kinetics of methanogenic bacteria (i.e., the saturation constant) may be strongly affected by temperature; i.e., it may be lower at thermophilic than at mesophilic temperature.

Plausible Causes for Inhibition

It is plausible to conclude that inhibition of the acetogenic and methanogenic activities during thermophilic digestion was caused by carbohydrate-, protein-, or lipid-degradation product(s). Carbohydrate degradation products under thermophilic conditions were not expected to be inhibitory to methane fermentation considering that cellulosic feeds reportedly digest better under thermophilic conditions than at mesophilic temperatures.[8,76] Comparison of steady-state data included in Table 73 on mesophilic and thermophilic digestion of the cellulosic fraction of municipal solid waste showed that there was no evidence of higher volatile acids accumulation during thermophilic fermentation.[77] Thermophilic methane yield was also higher than the mesophilic methane yield. Thus, products of thermophilic digestion of cellulosics do not seem to have any adverse effect on the acetogenic and

TABLE 72. COMPARISON OF STEADY-STATE METHANE YIELDS AND EFFLUENT VOLATILE ACIDS CONCENTRATION FROM MESOPHILIC AND THERMOPHILIC CFCSTR ACID-PHASE DIGESTION OF HANOVER PARK SLUDGE OPERATED AT ABOUT A 2-DAY HRT

	Mesophilic acid-phase		Thermophilic acid-phase
Two-phase system run no.	TP7M-M	TP7M-T	TP7T-T
HRT, days	1.9	1.9	2.1
Feed VS concentration, g/L	49.7	50.0	51.3
Methane yield, SCM/kg VS added	0.091	0.087	0.042
Volatile acids, mg/L			
Acetic	721	892	994
Propionic	728	849	723
Isobutyric	109	27	215
Butyric	160	159	198
Isovaleric	136	147	465
Valeric	61	27	5
Caproic	31	32	31
Total as acetic	1627	1826	2154

methanogenic organisms. In view of this, and considering that carbohydrate was degraded the least during thermophilic sludge digestion (Table 74) it may be inferred that products of protein and lipid degradation may have been inhibitory to thermophilic acetogens and methanogens.

Stability of Two-Phase Digestion

One major reason for digester instability is the accumulation of volatile acids and the concomittant drop in pH, both of which are inhibitory to the methanogens. The rate of pH drop and the magnitude of the final pH depend to a large extent on the buffer capacity of the digester contents; digester buffer capacity is mainly due to the bicarbonate alkalinity generated during the digestion process. Bicarbonate alkalinity increases with increases in gas (CO_2) and ammonia-nitrogen concentrations. Generally, the two-phase systems exhibited higher CO_2 and ammonia-nitrogen productions than those of the corresponding single-stage processes. In particular, the methane phases of the two-phase systems generated significantly higher bicarbonate alkalinities and buffer capacities than those of the single-stage processes, and this differential increased as the system HRT decreased (Tables 66, 67, and 68). For example, at a 15-day system HRT, the bicarbonate alkalinity of the meso-

TABLE 73. STEADY-STATE EFFLUENT VOLATILE ACIDS CONCENTRATIONS IN MESOPHILIC AND THERMOPHILIC SINGLE-STAGE CFCSTR DIGESTERS OPERATED WITH THE CELLULOSE FRACTION OF MUNICIPAL SOLID WASTE AT A 7-DAY HRT

	Mesophilic	Thermophilic	
HRT, days	7.3	7.1	7.1
Feed VS concentration, g/L	32.8	35.3	48.9
Methane yield, SCM/kg VS added	0.131	0.156	0.131
Volatile acids, mg/L			
Acetic	30	31	12
Propionic	360	132	124
Isobutyric	0	0	0
Butyric	0	0	1
Isovaleric	0	0	0
Valeric	0	3	14
Caproic	0	0	18
Total as acetic	330	139	130

TABLE 74. PROTEIN, CARBOHYDRATE, AND LIPID CONVERSIONS AT STEADY-STATE IN THERMOPHILIC CFCSTR METHANE-PHASE DIGESTERS FED WITH HANOVER PARK SEWAGE SLUDGE

Two-phase system run no.	TP15M-T	TP7M-T
Methane-phase HRT, days	13.0	5.5
Feed VS concentration, g/L	32.3	50.0
Reduction of organic components, %		
Crude protein	33.4	27.9
Carbohydrates	8.1	10.5
Lipids	64.1	63.9

meso two-phase system was 73% higher than the feed compared to a 53% increase for the single-stage process (Table 66). The corresponding increases at a 7-day HRT were 109% and 43%. At the very short system HRT of 3 days the bicarbonate alkalinity of the two-phase process increased by 441% compared with only 88% in the single-stage process. It is well accepted that the higher the buffer capacity of the culture the less vulnerable is the methanogenic culture to volatile acids accumulation and the more stable is the overall digestion process. The two-phase digestion process is more stable, and therefore, more reliable than single-stage digestion because a higher buffer capacity is maintained in the separated methane phase. The enhanced stability of the two-phase process relative to single-stage digestion increased by larger and larger amounts as the system HRT was decreased from 15 to 7 to 3 days.

SECTION 14

PERFORMANCE OF ACID-PHASE RUNS: PARAMETRIC-EFFECT STUDIES

EXPERIMENTAL RUNS

Six mesophilic and six thermophilic acid-phase runs were conducted with Hanover Park sewage sludge at HRT's of 2.0-2.4 and 1.3-1.5 days and at pH's of 7.0-7.1, 6.0-6.2, 5.5, and 5 (Table 75). Sodium hydroxide was used to control the pH at 7, whereas hydrochloric acid was used to control the pH at 6 or lower. All runs were conducted for a minimum duration of four HRT's. Most runs continued for a duration of about 10 HRT's. The maximum variability (coefficient of variation) of the culture pH and temperature were 4%. The variability of the digester HRT was between 2% and 13%. All runs were conducted with feed VS concentrations ranging between 46 and 54 g/L with an average concentration of about 50 g/L, which was the target VS consistency.

MESOPHILIC ACID-PHASE RUNS

pH Effects at a Two-Day HRT

The effects of pH on mesophilic acid-phase digester performance with Hanover Park sewage sludge feed are apparent from the data presented in Tables 76 through 78 and Figure 16. Consideration of the gas production and volatile acids production data shows that the optimum pH for acid-phase digestion of the feed sludge was between about 5.5 and 6.2. At an HRT of 2 days, protein and lipids reductions were 18% and 23% higher at a pH 5 than they were at pH 7; however, total carbohydrate reduction at pH's 7 and 5 were the same.

Hydrogen gas was not detected in any of the runs; consequently, inhibition of acetogenic organisms probably did not occur. Reduction of the total mass of carbohydrate-protein-lipid (ΣCPL) was higher at pH 5 than at pH 7.

pH Effects at an HRT of 1.3 Days

Data presented in Tables 79, 80, and 81 show that at an HRT of 1.3 days, acid-phase digester performance at pH 5 was worse than that at pH 7 when compared in terms of gas and volatile acids productions; and protein, carbohydrate and lipid reductions. These observations were opposite of those made during acid digester operation at a 2-day HRT. Apparently, a combination of low pH and low HRT adversely affected acid-phase digestion of sewage sludge under mesophilic conditions.

TABLE 75. ACTUAL STEADY-STATE OPERATING CONDITIONS FOR PARAMETRIC-EFFECTS CFCSTR ACID-PHASE DIGESTERS FED WITH HANOVER PARK SLUDGE*

Run no.	Digester no.	Total run duration, days	Steady-state duration, days	Culture temperature, °C	Mean culture pH	Mean HRT, days	Mean loading rate, kg VS/m^3-day	Feed total solids concentration, g/L	Feed volatile solids concentration, g/L
Mesophilic									
AP2M7	334	63	40	35.1 (1)**	6.99 (3)	2.1 (12)	23.43 (11)	69.8	48.3
AP2M6	334	20	20	35.7 (1)	6.17 (4)	2.4 (20)	22.20 (25)	65.3	52.4
AP2M5.5	334	9	9	35.8 (1)	5.47 (2)	2.2 (6)	23.40 (6)	65.5	50.3
AP2M5	334	13	13	35.5 (1)	5.02 (1)	2.1 (5)	24.62 (5)	67.3	51.5
AP1.3M7	334	30	22	35.2 (1)	69.6 (4)	1.3 (10)	34.08 (10)	60.1	46.0
AP1.3M5	334	13	12	35.7 (1)	5.04 (2)	1.5 (10)	32.76 (10)	68.4	48.5
Thermophilic									
AP2T7	335	98	79	54.3 (3)	7.08 (2)	2.1 (13)	25.48 (12)	76.1	52.8
AP2T6	335	11	9	56.4 (2)	5.95 (3)	2.1 (2)	24.25 (2)	65.5	50.0
AP2T5.5	335	18	17	54.4 (4)	5.53 (2)	2.0 (16)	26.13 (21)	69.2	53.6
AP2T5	335	20	11	54.8 (1)	5.07 (2)	2.1 (3)	24.03 (3)	65.3	50.0
AP1.3T7	335	56	39	55.4 (1)	7.04 (1)	1.3 (7)	36.02 (12)	62.7	47.9
AP1.3T5	335	15	14	55.2 (1)	4.99 (2)	1.3 (7)	38.74 (6)	67.5	51.1

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* Data reported are the means of all data collected during the steady-state period. Batch numbers for the various runs are shown in Appendix Table F-1.

** Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of standard deviation to the mean.

TABLE 76. EFFECT OF pH ON STEADY-STATE GAS PRODUCTIONS FROM MESOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 2-DAY HRT[a]

Culture pH	7.0	6.2	5.5	5.0
Run no.	AP2M7	AP2M6	AP2M5.5	AP2M5
Operation				
Feed VS concentration,[b] mg/L	48,270	52,390	50,310	51,460
HRT, days	2.1	2.4	2.2	2.1
Loading, kg VS/m^3-day	23.43	22.20	23.40	24.62
pH-control chemical (2.5N)	NaOH	HCl	HCl	HCl
pH-control dosage, meq/L feed	32.9	23.4	46.0	80.9
Performance				
Total gas yield, SCM/kg VS added	0.049	0.133	0.093	0.058
	(22)[c]	(14)	(12)	(23)
Methane yield, SCM/kg VS added	0.035	0.071	0.048	0.029
	(21)	(14)	(12)	(24)
Gas composition, mol %				
Hydrogen	--	--	--	0.0
Methane	71.4	53.2	51.7	50.1
Carbon Dioxide	27.0	46.6	48.0	49.2
Nitrogen	1.6	0.2	0.3	0.7
Total gas production rate, SCM/m^3-day	1.124	2.887	2.166	1.424
	(17)	(13)	(8)	(21)
Methane production rate, SCM/m^3-day	0.804	1.542	1.120	0.713
	(17)	(16)	(8)	(23)

[a] Data reported are means of all data collected during the steady-state portion of the run.

[b] Feed VS concentrations are the weighted averages of the various feed slurry concentrations.

[c] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of the standard deviation to the mean.

TABLE 77. EFFECT OF pH ON STEADY-STATE EFFLUENT QUALITITES OF MESOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 2-DAY HRT*

Culture pH	7.0	6.2	5.5	5.0
Run no.	AP2M7	AP2M6	AP2M5.5	AP2M5
Alkalinities, mg/L as $CaCO_3$				
Total	8160	--	--	2520
Bicarbonate	6963	--	--	13
Volatile acids, mg/L				
Acidic	662	1145	1465	1087
Propionic	646	1113	1223	740
Isobutyric	100	128	160	176
Butyric	118	335	547	524
Isovaleric	195	271	1002	932
Valeric	13	109	185	282
Caproic	2	11	39	5
Total as acetic	1457	2592	3657	2880
Ethanol, mg/L	3	0	0	79
Nitrogen, mg/L				
Ammonia-N	505	--	--	923
Organic-N	1638	--	--	2150
Chemical oxygen demand, mg/L				
Total	68,480	--	--	--
Filtrate	5064	--	--	--
Solids, mg/L				
TS	62,120	--	--	59,290
VS	39,650	--	--	44,590
TSS	55,080	--	--	--
VSS	40,280	--	--	--
Organic components, mg/L				
Crude protein	10,238	--	--	13,438
Carbohydrates	7851	--	--	10,561
Lipids	14,444	--	--	10,988

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* Data reported are means of one or more determinations made during steady-state period.

TABLE 78. EFFECT OF pH ON STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF MESOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 2-DAY HRT[a]

Culture pH	7.0	6.2	5.5	5.0
Run no.	AP2M7	AP2M6	AP2M5.5	AP2M5
VS reduction, %				
MOP_{16}[b]	15.3	--	--	5.2
Wt-of-gas basis[c]	5.2	16.0	11.6	3.5
VSS reduction	11.8	--	--	--
COD (total) reduction, %	11.9	--	--	--
Organic component reduction, %				
Crude protein	20.0	--	--	24.7
Carbohydrates	9.3	--	--	9.4
Lipids	21.7	--	--	27.2
ΣCPL[d]	18.4	--	--	21.6

[a] Data reported are means of one or more determinations made during steady-state period.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 X (VS_i0 - VS_o)/[VS_i - (VS_i X VS_o)]$.

[c] These VS reductions were calculated according the following formula: $VS_R = 100 X$ (wt of product gases)/(wt of VS fed).

[d] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

HRT Effect

Comparing mesophilic acid-digester performances at pH 7 at HRT's of 2.0 and 1.3 days, gas and volatile acids productions, and protein, carbohydrate and lipid reductions were significantly higher than at the lower HRT (Table 82). Similar comparison of acid-phase digester performances at pH 5 showed that whereas gas and acid productions at HRT's of 2.0 and 1.3 days were about the same, protein and lipid reductions were lower at the lower HRT (Table 83); carbohydrate conversion at 1.3-day HRT, however, was higher than that at a 2-day HRT.

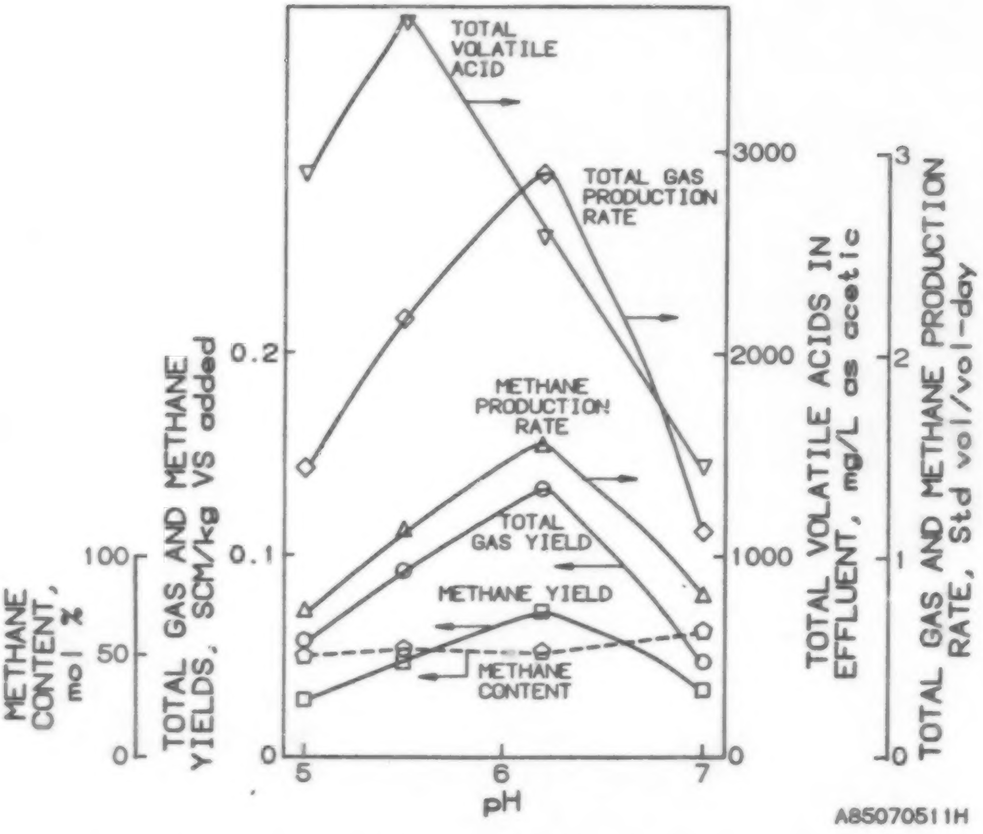

Figure 16. Effect of pH on mesophilic acid-phase digestion of Hanover Park sewage sludge at an HRT of about 2.2 days and a loading rate of about 23 kg VS/m^3-day.

TABLE 79. EFFECT OF pH ON STEADY-STATE GAS PRODUCTIONS FROM MESOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 1.3-DAY HRT[a]

Culture pH	7.0	5.0
Run no.	AP1.3M7	AP1.3M5
Operation		
Feed VS concentration,[b] mg/L	46,000	48,490
HRT, days	1.3	1.5
Loading, kg VS/m^3-day	34.08	32.76
pH-control chemical (2.5N)	NaOH	HCl
pH-control dosage, meq/L feed	69.2	52.8
Performance		
Total gas yield, SCM/kg VS added	0.085	0.062
	(12)[c]	(23)
Methane Yield, SCM/kg VS added	0.056	0.031
	(16)	(19)
Gas composition, mol %		
Hydrogen	--	0.04
Methane	65.3	50.3
Carbon dioxide	34.1	48.9
Nitrogen	0.6	0.8
Total gas production rate, SCM/m^3-day	2.889	2.075
	(9)	(15)
Methane production rate, SCM/m^3-day	1.891	1.046
	(15)	(12)

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[a] Data reported are means of all data collected during the steady-state portion of the run.

[b] Feed VS concentrations are the weighted averages of the various feed slurry concentrations.

[c] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of the standard deviation to the mean.

TABLE 80. EFFECT OF pH ON STEADY-STATE EFFLUENT QUALITIES OF MESOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 1.3-DAY HRT

Run no.	AP1.3M7	AP1.3M5
Culture pH	7.0	5.0
Alkalinities, mg/L as $CaCO_3$		
Total	7950	5660
Bicarbonate	4116	3872
Volatile acids, mg/L		
Acetic	2498	1140
Propionic	1641	963
Isobutyric	258	148
Butyric	536	565
Isovaleric	382	485
Valeric	91	317
Caproic	0	22
Total as acetic	4648	2889
Ethanol, mg/L	0	22
Nitrogen, mg/L		
Ammonia-N	1008	757
Organic-N	1916	2084
Chemical oxygen demand, mg/L		
Total	75,480	--
Filtrate	8973	--
Solids, mg/L		
TS	51,060	60,890
VS	35,410	43,200
TSS	38,370	--
VSS	31,620	--
Organic components, mg/L		
Crude protein	11,975	13,025
Carbohydrates	7114	10,849
Lipids	9202	9176

* Data reported are means of one or more determinations made during the steady-state period.

TABLE 81. EFFECT OF pH ON STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF MESOPHILIC CFCSTR DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 1.3-DAY HRT[a]

Run no.	AP1.3M7	AP1.3M5
Culture pH	7.0	5.0
VS reduction, %		
MOP_{16}[b]	27.4	0.0
Wt-of-gas basis[c]	9.6	8.1
VSS reduction	19.3	--
COD (total) reduction, %	7.5	--
Organic reductions, %		
Crude protein	24.6	10.8
Carbohydrates	42.1	20.4
Lipids	21.4	6.6
ΣCPL[d]	29.0	13.2

[a] Data reported are means of one or more determinations made during the steady-state period.

[b] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[c] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[d] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

THERMOPHILIC ACID-PHASE RUNS

pH Effects at a Two-Day HRT

Tables 84, 85, 86 and Figure 17 present experimental data collected to delineate the effects of pH on thermophilic acid-phase digestion of Hanover Park sewage sludge at an HRT of 2 days. Apparently, optimum thermophilic acid

TABLE 82. COMPARISON OF STEADY-STATE PERFORMANCES OF MESOPHILIC AND THERMOPHILIC ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT pH 7

HRT, days	2	2	1.3	1.3
Run no.	AP2M7	AP2T7	AP1.3M7	AP1.3T7
Culture temperature	Mesophilic	Thermophilic	Mesophilic	Thermophilic
NaOH dosage, meq/L feed	32.9	30.5	69.2	17.6
Total gas yield, SCM/kg VS added	0.049	0.014	0.085	0.046
Methane yield, SCM/kg VS-day	0.035	0.008	0.056	0.026
Methane content, mol %	71.4	58.6	65.3	57.2
Total gas production rate, SCM/m^3-day	1.124	0.345	2.889	1.615
Effluent volatile acids, mg/L as acetic	1457	3220	4648	4184
VS reduction, %				
MOP_{16}[a]	15.3	3.7	27.4	5.9
Wt-of-gas basis[b]	5.2	1.6	9.6	5.9
Carbon-in-gas basis[c]	4.4	1.2	7.9	4.2
VSS reduction, %	11.8	2.8	19.3	17.2
COD (total) reduction, %	11.9	1.8	7.5	10.5
Organic reduction, %				
Crude protein	20.0	42.2	24.6	34.4
Carbohydrates	9.3	48.9	42.1	22.8
Lipids	21.7	31.1	21.4	15.2
ΣCPL[d]	18.4	40.6	29.0	25.2

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[a] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[c] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (1.84 X wt of carbon in product gas)/(wt of VS fed).

[d] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

TABLE 83. COMPARISON OF STEADY-STATE PERFORMANCES OF MESOPHILIC AND THERMOPHILIC ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT pH 5

HRT, days	2	2	1.3	1.3
Run no.	AP2M5	AP2T5	AP1.3M5	AP1.3T5
Culture temperature	Mesophilic	Thermophilic	Mesophilic	Thermophilic
HCl dosage, meq/L feed	80.9	69.5	52.8	58.3
Total gas yield, SCM/kg VS added	0.058	0.031	0.062	0.017
Methane yield, SCM/kg VS-day	0.029	0.015	0.031	0.009
Methane content, mol %	50.1	49.1	50.3	50.6
Total gas production rate, SCM/m^3-day	1.424	0.745	2.075	0.663
Effluent volatile acids, mg/L as acetic	2880	3494	2889	3222
VS reduction, %				
MOP_{16}[a]	5.2	7.1	0.0	4.8
Wt-of-gas basis[b]	3.5	4.0	8.1	2.2
Carbon-in-gas basis[c]	5.4	2.8	5.9	1.5
Organic reduction, %				
Crude protein	24.7	22.9	10.8	17.0
Carbohydrates	9.4	13.8	20.4	8.4
Lipids	27.2	28.0	6.6	23.0
ΣCPL[d]	21.6	22.2	13.2	16.8

[a] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[c] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (1.84 X wt of carbon in product gas)/(wt of VS fed).

[d] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

TABLE 84. EFFECT OF pH ON STEADY-STATE GAS PRODUCTIONS FROM THERMOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 2-DAY HRT[a]

Culture pH	7.1	6.0	5.5	5.1
Run no.	AP2T7	AP2T6	AP2T5.5	AP2T5
Operation				
Feed VS concentration,[b] mg/L	52,750	49,960	53,560	49,980
HRT, days	2.1	2.1	2.0	2.1
Loading, kg VS/m^3-day	25.48	24.25	26.13	24.03
pH-control chemical (2.5N)	NaOH	HCl	HCl	HCl
pH-control dosage, meq/L feed	30.5	33.2	58.6	69.5
Performane				
Total gas yield, SCM/kg VS added	0.014	0.052	0.024	0.031
	(24)[c]	(9)	(21)	(11)
Methane yield, SCM/kg VS added	0.008	0.024	0.009	0.015
	(22)	(13)	(40)	(11)
Gas composition, mol %				
Hydrogen	--	--	--	0.2
Methane	58.6	46.0	38.4	49.1
Carbon dioxide	38.6	53.2	57.7	49.2
Nitrogen	2.8	0.8	3.9	1.5
Total gas production rate, SCM/m^3-day	0.345	1.251	0.499	0.745
	(22)	(9)	(25)	(10)
Methane production rate, SCM/m^3-day	0.202	0.575	0.230	0.366
	(20)	(12)	(37)	(10)

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[a] Data reported are means of all data collected during the steady-state portion of the run.

[b] Feed VS concentrations are the weighted averages of the various feed slurry concentrations.

[c] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of the standard deviation to the mean.

TABLE 85. EFFECT OF pH ON STEADY-STATE EFFLUENT QUALITIES OF THERMOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 2-DAY HRT*

Culture pH	7.1	6.0	5.5	5.1
Run no.	AP2T7	AP2T6	AP2T5.5	AP2T5
Alkalinities, mg/L as $CaCO_3$				
Total	8900	--	--	3000
Bicarbonate	5680	--	--	1255
Volatile acids, mg/L				
Acetic	1445	2171	1944	1976
Propionic	986	1219	1229	845
Isobutyric	336	326	220	177
Butyric	461	580	833	376
Isovaleric	686	652	412	734
Valeric	50	89	144	43
Caproic	0	24	19	1
Total as acetic	3220	4223	3994	3494
Ethanol, mg/L	11	0	24	86
Nitrogen, mg/L				
Ammonia-N	980	--	--	959
Organic-N	1370	--	--	2204
Chemical oxygen demand, mg/L				
Total	81,120	--	--	--
Filtrate	10,820	--	--	--
Solids, mg/L				
TS	72,245	--	--	57,710
VS	49,745	--	--	43,260
TSS	67,030	--	--	--
VSS	46,430	--	--	--
Organic components, mg/L				
Crude protein	7870	--	--	13,775
Carbohydrates	6934	--	--	9943
Lipids	9735	--	--	10,539

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* Data reported are means of one or more determinations made during the steady-state period.

TABLE 86. EFFECT OF pH ON STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF THERMOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 2-DAY HRT[a]

Culture pH	7.1	6.0	5.5	5.1
Run no.	AP2T7	AP2T6	AP2T5.5	AP2T5
VS reduction, %				
MOP_{16}[b]	3.7	--	--	7.1
Wt-of-gas basis[c]	1.6	7.0	3.1	4.0
VSS reduction	2.8	--	--	--
COD (total) reduction, %	1.8	--	--	--
Organic component reduction, %				
Crude protein	42.2	--	--	22.9
Carbohydrates	48.9	--	--	13.8
Lipids	31.1	--	--	28.0
ΣCPL[d]	40.6	--	--	22.2

[a] Data reported are means of one or more determinations made during steady-state period.

[b] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (VS_i0 - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[c] These VS reductions were calculated according the following formula: $VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[d] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

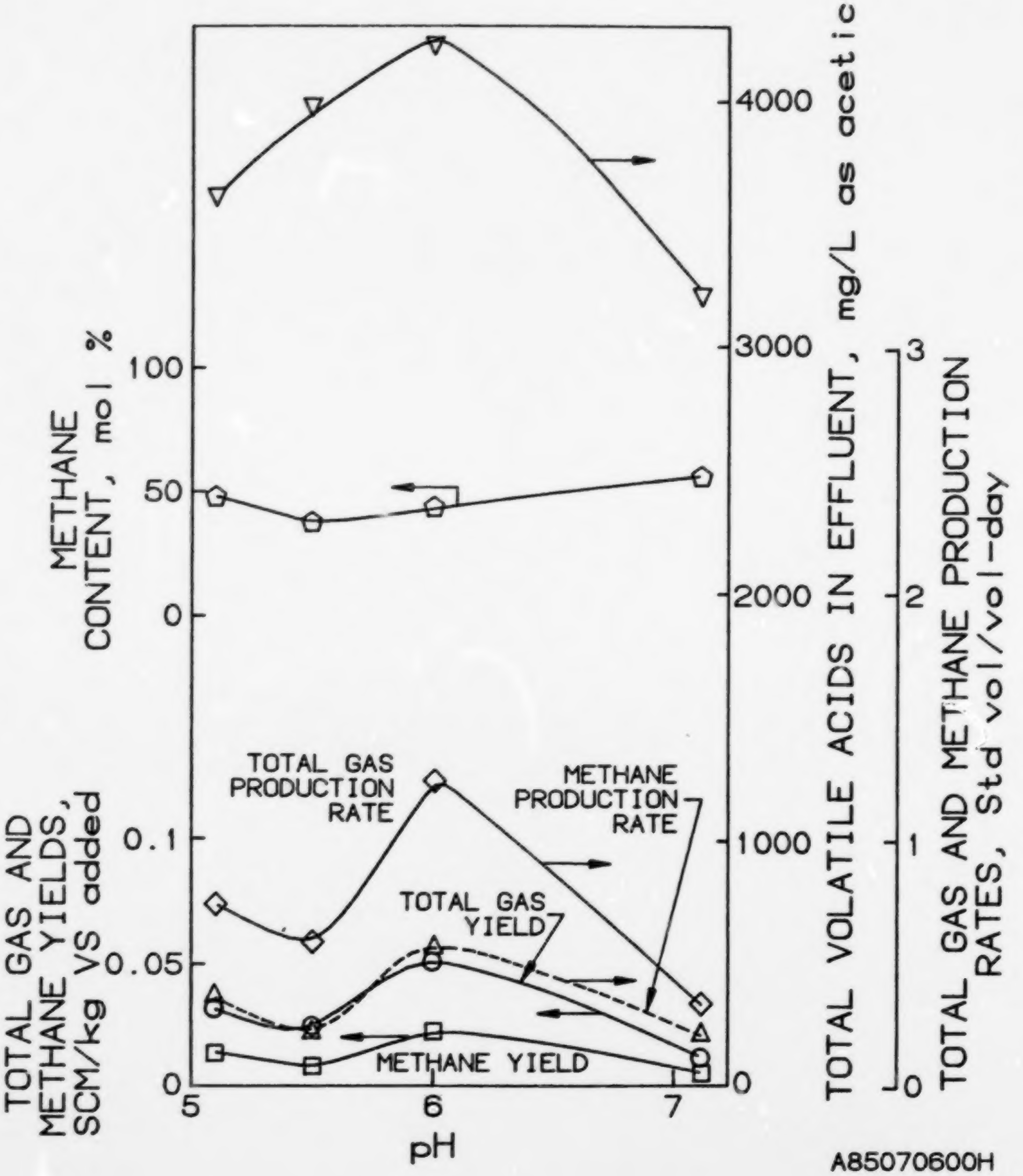

Figure 17. Effect of pH on thermophilic acid-phase digestion of Hanover Park sewage sludge at an HRT of about 2.1 days and a loading rate of about 25 kg VS/m^3-day.

digester performance was obtained at a pH of 6. Protein, carbohydrate, and lipid degradations were higher at pH 7 than at pH 5; volatile acids production at these two pH's were about the same. The depressed hydrolytic activities at pH 5 may have resulted due to the presence of hydrogen in the gas phase at pH 5. The presence of hydrogen indicated that the production of this gas was greater than its removal by the hydrogen-utilizing methanogens. This observation could be indicative of the fact that hydrogen-oxidizing thermophilic methane bacteria are inhibited at pH 5.

Crude protein and carbohydrate reduction were much lower at pH 5 than they were at pH 7; however, lipid degradation were about the same at these two pH's.

pH Effect at an HRT of 1.3 Days

Data reported in Tables 87, 88, and 89 show that at a 1.3-day HRT gas and methane production at pH 5 were significantly lower than those at pH 7. These results suggest that the activities of the syntrophic methane formers were depressed under thermophilic conditions at a pH of 5. The presence of hydrogen in the gas phase at pH 5 attests to this hypothesis.

Table 88 indicates that protein, carbohydrate, and SCPL reductions were lower, but lipid conversion was higher at pH 5 than they were at pH 7. Volatile acids production at pH 7 was significantly greater than that at pH 5.

HRT Effect

Comparing thermophilic acid-digester performances at pH 7 at HRT's of 2.0 and 1.3 days, gas and volatile acids productions were higher at 1.3 days, although organic reductions were lower at the lower HRT (Table 82). Similar comparison at pH 5 showed that the thermophilic acid-digester performance deteriorated significantly when the HRT was decreased from 2.0 to 1.3 days (Table 83). At pH 5 gas and volatile acids productions and organic reductions were lower at a 1.3-day HRT than they were at a 2-day HRT.

EFFECT OF TEMPERATURE ON ACID-PHASE DIGESTION

Consideration of data presented in Tables 82 and 83 indicated that at a 2-day HRT, the thermophilic acid digester exhibited higher liquefaction-acidification efficiency than the mesophilic acid digester at both pH 7 and pH 5. The same was also true at an HRT of 1.3 days at pH 5, but not at pH 7. At pH 7 and a 1.3-day HRT the mesophilic acid digester exhibited slightly higher volatile acid production, and carbohydrate and lipid reductions than the thermophilic acid digester. It is noteworthy that gas and methane productions from the thermophilic acid digester were lower than those of the mesophilic acid digester under all operating conditions. Thus, the activities of the thermophilic syntrophic methanogens were considerably lower than those of their mesophilic counterparts. It is speculated that thermophilic metabolites of sludge organics could be instrumental in depressing the activities of the syntrophic methanogens.

TABLE 87. EFFECT OF pH ON STEADY-STATE GAS PRODUCTIONS FROM THERMOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 1.3-DAY HRT[a]

Culture pH	7.0	5.0
Run no.	AP1.3T7	AP1.3T5
Operation		
Feed VS concentration,[b] mg/L	47,900	51,140
HRT, days	1.3	1.3
Loading, kg VS/m^3-day	36.02	38.74
pH-control chemical (2.5N)	NaOH	HCl
pH-control dosage, meq/L feed	17.6	58.3
Performance		
Total gas yield, SCM/kg VS added	0.046 (24)[c]	0.017 (13)
Methane yield, SCM/kg VS added	0.026 (25)	0.009 (14)
Gas composition, mol %		
Hydrogen	--	0.4
Methane	57.2	50.6
Carbon dioxide	41.8	45.6
Nitrogen	1.0	2.4
Total gas production rate, SCM/m^3-day	1.615 (15)	0.664 (14)
Methane production rate, SCM/m^3-day	0.923 (16)	0.335 (14)

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[a] Data reported are means of all data collected during the steady-state portion of the run.

[b] Feed VS concentrations are the weighted averages of the various feed slurry concentrations.

[c] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of the standard deviation to the mean.

TABLE 88. EFFECT OF pH ON STEADY-STATE EFFLUENT QUALITIES OF THERMOPHILIC CFCSTR ACID-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 1.3-DAY HRT*

Culture pH	7.0	5.0
Run no.	AP1.3T7	AP1.3T5
Alkalinities, mg/L as $CaCO_3$		
Total	8625	4915
Bicarbonate	5079	2227
Volatile acids, mg/L		
Acetic	1756	1946
Propionic	1306	786
Isobutyric	416	162
Butyric	689	405
Isovaleric	984	312
Valeric	66	101
Caproic	0	18
Total as acetic	4184	3222
Ethanol, mg/L	0	85
Nitrogen, mg/L		
Ammonia-N	1308	916
Organic-N	1582	2398
Chemical oxygen demand, mg/L		
Total	68,400	--
Filtrate	10,070	--
Solids, mg/L		
TS	53,410	60,860
VS	40,340	45,170
TSS	36,910	—
VSS	30,400	--
Organic components, mg/L		
Crude protein	9885	14,988
Carbohydrates	9036	10,595
Lipids	9451	11,466

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* Data reported are means of one or more determinations made during the steady-state period.

TABLE 89. EFFECT OF pH ON STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF THERMOPHILIC CFCSTR DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 1.3-DAY HRT[a]

Culture pH	7.0	5.0
Run no.	AP1.3T7	AP1.3T5
VS reduction, %		
MOP_{16}[b]	5.9	4.8
Wt-of-gas basis[c]	5.9	2.2
VSS reduction	17.2	--
COD (total) reduction, %	10.5	--
Organic reductions, %		
Crude protein	34.4	17.0
Carbohydrates	22.8	8.4
Lipids	15.2	23.0
ΣCPL[d]	25.2	16.8

[a] Data reported are means of one or more determinations made during the steady-state period.

[b] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[c] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[d] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

ANALYSIS OF VARIANCE AND STATISTICAL INFERENCE

As mentioned in a previous section, the acid-phase parametric-effect studies were conducted according to a factorial experiment design to explore the effect of the control (or treatment) variables (or factors) of culture temperature, pH, and HRT on such observable response variables or variates as gas yield, gas production rate, volatile acid production, and reductions of major organic components (e.g., carbohydrates, proteins and lipids) at steady state. Temperature was set at levels of 35°C and 55°C, pH at 5 and 6, and HRT at 2 days and 1.3 days. There were eight treatments (or runs), each at a different combination of the control variables. Two to 12 replicates of each variate at steady state were considered for each run. The results of analysis of variance (ANOVA) of the steady-state acid-phase digestion data are shown in Tables 90 and 91. The ANOVA results may be interpreted as follows:

- The culture pH appeared to have a strong effect on carbohydrate, crude protein, lipid, and ΣCPL (sum of the carbohydrate, protein and lipid masses) reduction efficiencies almost regardless of the levels of the other variables (temperature and HRT in this case). The effect of increasing the culture pH from 5 to 7 was to increase carbohydrate, protein, lipid and ΣCPL reductions by about 19, 14, 14 and 23 percentage points, respectively. It should be noted, however, that the above ANOVA analysis does not identify the existence of a probable optimum pH lying between the values of 5 and 7.

- Increases in culture temperature and digester HRT both tended to increase carbohydrate reduction; however, the effects of these two control variables cannot be viewed independently because of the large interaction effect.

- Increase in HRT tended to increase protein reduction, but the HRT effect was influenced strongly by the culture pH.

- Temperature had no significant effect on lipid reduction.

- Increase in HRT from 1.3 to 2 days increased lipid reduction by about 13 percentage points. The HRT effect was not influenced by pH or temperature.

- Increases in temperature and HRT increased ΣCPL reduction, but effects of these control variables cannot be separated.

- The digester HRT, temperature, and culture pH, each independently influenced the acid-digester gas yield. The gas yield decreased as the digester HRT and culture temperature increased, and it increased as the culture pH was increased.

- The digester temperature independently acted on the acid-phase gas production rate, which decreased substantially (by 1.04 vol/culture vol-day) as the temperature was increased from 35°C to 55°C.

- The effects of pH and HRT on acid-digester gas production could not be separated because of the large pH-HRT interaction effect. However, an increase in pH tended to increase the gas production rate. On the other hand, an increase in HRT decreased gas production rate.

- An increase in HRT increased the methane content of the acid-digester gas, regardless of the levels of pH and temperature. Increases in pH and temperature seemed to have the effect of increasing the methane content, but the effects of these variables cannot be viewed separately.

TABLE 90. RESULTS OF ANALYSIS OF VARIANCE (ANOVA) OF ACID-PHASE DIGESTION STEADY-STATE DATA TO ASSESS THE EFFECTS OF THE CONTROL VARIABLES OF CULTURE TEMPERATURE, pH, AND HRT ON REDUCTIONS OF CARBOHYDRATES, PROTEIN, AND LIPIDS

Response variable	Source of variance	Sum of squares	Degrees of freedom	Mean square	Computed F	F-critical $\alpha = 0.05$	Control variable	Main effects	95% confidence limits*	Interactions	Interaction effect	95% confidence limits*
Carbohydrate reduction, %	Mean	8710.66	1	--	--	--	Temp, °C	11.47	5.06 to 17.88	Temp-pH	7.60	1.19 to 14.01
	Treatments	4699.13	7	671.30	--	--	pH	19.48	13.07 to 25.89	Temp-HRT	18.98	12.57 to 25.39
	Error	437.90	11	39.81	16.86	3.01	HRT, days	6.31	-0.09 to 12.72	pH-HRT	1.40	-5.00 to 7.81
Protein reduction, %	Mean	9838.09	1	--	--	--	Temp, °C	11.30	9.50 to 13.11	Temp-pH	6.05	4.24 to 7.86
	Treatments	1204.40	7	172.06	--	--	pH	13.92	12.11 to 15.73	Temp-HRT	-0.02	-1.83 to 1.78
	Error	15.10	7	2.16	79.78	3.79	HRT, days	7.68	5.87 to 9.49	pH-HRT	-5.71	-7.52 to -3.90
Lipid reduction, %	Mean	5572.35	1	--	--	--	Temp, °C	1.43	-2.88 to 5.73	Temp-pH	-4.80	-9.11 to -0.49
	Treatments	500.04	7	71.43	--	--	pH	14.17	9.87 to 18.48	Temp-HRT	5.50	1.20 to 9.81
	Error	15.68	3	5.23	13.67	8.89	HRT, days	13.16	8.85 to 17.46	pH-HRT	3.85	-0.45 to 8.16
ΣCPL† reduction, %	Mean	35,397.30	1	--	--	--	Temp, °C	11.67	10.90 to 12.44	Temp-pH	4.48	3.71 to 5.25
	Treatments	4099.62	7	585.66	--	--	pH	23.30	22.54 to 24.07	Temp-HRT	15.59	14.82 to 16.36
	Error	79.62	43	1.85	316.29	2.24	HRT, days	17.84	17.07 to 18.60	pH-HRT	6.22	5.45 to 6.98

* If zero is contained within the confidence interval, it indicates that the effect is not significant at the 5% level.

† ΣCPL means the sum of the masses of carbohydrate, crude protein, and lipids.

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TABLE 91. RESULTS OF ANALYSIS OF VARIANCE (ANOVA) OF ACID-PHASE DIGESTION STEADY-STATE DATA TO ASSESS THE EFFECTS OF THE CONTROL VARIABLES OF CULTURE TEMPERATURE, pH, AND HYDRAULIC RESIDENCE TIME (HRT) ON TOTAL GAS YIELD, GAS PRODUCTION RATE, AND METHANE CONTENT

Response variable	Source of variance	Sum of squares	Degrees of freedom	Mean square	Computed F	F-critical $\alpha = 0.05$	Control variable	Main effects
Total gas yield, SCM/kg VS added	Mean	0.0989	1	--	--	--	Temp, °C	-0.0369
	Treatments	0.0247	7	0.0035	--	--	pH	0.0063
	Error	0.0016	40	0.000039	90.60	2.25	HRT, days	-0.0148
Total gas production rate, SCM/m^3-day	Mean	88.702	1	--	--	--	Temp, °C	-1.036
	Treatments	29.216	7	4.174	--	--	pH	0.266
	Error	1.070	40	0.027	155.999	2.25	HRT, days	-0.901
Methane content, mol %	Mean	128,460.00	1	--	--	--	Temp, °C	8.55
	Treatements	2038.37	7	291.20	--	--	pH	18.28
	Error	235.21	32	7.92	36.80	2.32	HRT, days	29.01

Response variable	95% confidence limits*	Interactions	Interaction effect	95% confidence limits*
Total gas yield, SCM/kg VS added	-0.0406 to -0.0333	Temp-pH	-0.0007	-0.0044 to 0.0029
	0.0027 to 0.0099	Temp-HRT	0.0059	0.0026 to 0.0095
	-0.0184 to -0.0112	pH-HRT	-0.0198	-0.0234 to -0.0162
Total gas production rate, SCM/m^3-day	-0.132 to -0.941	Temp-pH	0.011	-0.085 to 0.106
	0.171 to 0.362	Temp-HRT	0.307	0.212 to 0.402
	-0.997 to -0.806	pH-HRT	-0.616	-0.712 to -0.521
Methane content, mol %	6.74 to 10.36	Temp-pH	13.57	11.76 to 15.38
	16.47 to 20.09	Temp-HRT	4.92	3.11 to 6.73
	27.20 to 30.82	pH-HRT	15.09	13.28 to 16.9

* If zero is contained within the confidence interval, it indicates that the effect is not significant at the 5% level.

- Increases in temperature, pH and HRT increased volatile acids productions by acid-phase digestion, but the independent effects of these variables cannot be ascertained separately.

The above interpretations of the ANOVA analysis suggested that enhanced hydrolysis of the major organic components of sludge may not be achieved at the lower pH, HRT, and temperature of 5, 1.3 days, and 35°C, respectively. The analysis also suggested that the acidification process, which follows hydrolysis, was also not the most efficient at pH 5, 35°C, and a 1.3-day HRT. According to the ANOVA results, hydrolysis and acidification efficiencies were higher at the thermophilic temperature and at a pH higher than 5. This is in agreement with discussions presented in the earlier sections which indicated that a pH of about 6 would be optimum for these reactions; this pH appeared to be optimum also for maximized gas production rate and gas yield. The statistical analysis indicated that a thermophilic temperature and a pH 5 decreased gas yield and gas production rate; this inference suggested that the syntrophic methane formers were probably inhibited under these operating conditions. Similar conclusions were also drawn from the results of the single-stage CFCSTR and two phase digestion studies.

SECTION 15

ADVANCED TWO-PHASE DIGESTION TESTS: APPLIED STUDIES

TWO-PHASE PROCESS IMPROVEMENT WITH NOVEL UPFLOW REACTORS

As discussed in detail in Section 4 of this report, the application of a high-SRT reactor — that is, a reactor in which the SRT is considerably longer than the HRT — has the effect of significantly enhancing the substrate conversion efficiency relative to that achieved with a CFCSTR digestion reactor in which SRT equals HRT. It is also important to note that there are only a few anaerobic reactor designs which can promote efficient digestion of particulate solids and simultaneously effect prolonged retention of microbial and substrate solids to exhibit high SRT's. Bioreactors that provide efficient solids retention may also experience short-circuiting, creation of dead zones, and accumulation of unreacted solids. Thus, the benefits of having high SRT's could be readily neutralized by the detrimental effects of short-circuiting, dead zones, and the accumulation of biologically inert solids if an appropriate reactor design is not utilized. Since little work has been done on characterizing novel reactor performance, guidelines for the design of appropriate high-SRT reactors for acid- and methane-phase digestion of wastewater sludge were not available. Consequently, the reactor development approach utilized in the applied studies was an empirical one and involved the design, construction, and testing of an innovative biodigester that incorporated structural and operating features which were expected to promote solids retention, and to exhibit higher sludge stabilization efficiencies than the CFCSTR digester.

The innovative digesters utilized in the applied studies were of the upflow type. This particular type of bioreactor was used for acid- and methane-phase digestion of a difficult-to-treat wastewater sludge in a previous research project, and exhibited better performance than the CFCSTR digesters.[23] The upflow digesters used in the applied studies were improved versions of the earlier design which provided for a vertical standpipe feed inlet and a canopy solids deflector directly above it. As described in detail in Section 6, the acid digester (Digester 338) incorporated the above mentioned features, and in addition, included vertical baffles and hoppered bottoms to facilitate the containment of floating scum and the controlled withdrawal of a volatile acids-rich underflow stream that was pumped to the methane digester. The upflow methane digester (Digester 339) included static horizontal ring baffles and rotating impellors between them to minimize short circuiting, and to achieve plug-flow characteristics. Provisions were made to recycle methane digester effluents to the acid digester to promote hydrogen removal.

Optimum Operating Conditions for Two-Phase Digestion

Information compiled from the process-comparison and the parametric-effect acid-phase runs (Sections 11 through 14) was used to select the optimum operating conditions for the advanced two-phase run. A mesophilic temperature was preferred to the thermophilic temperature which was found to inhibit acetogenic and methanogenic conversions (see Section 13). However, thermophilic digestion studies were also conducted after termination of the mesophilic run to investigate the feasibility of thermophilic two-phase digestion with high-SRT digesters. An HRT of 7 days and a feed VS concentration of about 50 g/L were selected for the advanced two-phase run because the process-comparison studies indicated this HRT-feed-VS concentration combination to be optimum from the viewpoint of methane production and solids stabilization. HRT's of about 2 and 5 days were selected for the acid- and methane-phase digesters. Based on the information collected from the parametric-effect acid-phase runs, a pH of about 6.5 was regarded as optimum for sludge acidogenesis. Since this pH is "naturally" obtained during sludge acidogenesis, the need for pH control with external chemicals entailing added operating cost was ruled out.

Mesophilic Two-Phase Digestion With Upflow Reactors

A two-phase system comprised of the upflow acid- and methane-phase digesters described above was operated under the optimum digestion conditions delineated in the foregoing section. The upflow two-phase system was operated for about 2.5 months at a mesophilic temperature and at a system HRT of about 7.5 days (Table 92). Steady-state operating conditions and performance characteristics of the upflow acid and methane digesters are detailed in Tables 92, 93, and 94.

Conventional CFCSTR digesters are generally operated for about three HRT's at steady state once constant performance levels are reached. Since no such criterion was available for unconventional reactors, the upflow acid- and methane-phase digesters were operated for about 36 and 13 HRT's, respectively, to ensure the achievement of steady state. The variabilities of total gas and methane yields, production rates, and volatile acid productions during a 37-day period of operation of the upflow two-phase system were comparable to those of the CFCSTR two-phase and the CFCSTR single-stage runs at steady state (see Tables 93 and 94). These variabilities were indicative of the fact that the upflow system was at steady state — variabilities of acid digester gas yields and methane digester effluent volatile acids were rather high due to the low levels of these parameters and the associated inaccuracies and poor precision of measurements. Achievement of a steady-state operation of the upflow digesters was also indicated by the constancy of the volatile acids profiles in the acid- and methane-phase digesters (Table E-2).

The upflow two-phase system treatment resulted in a methane yield of 0.352 SCM/kg VS (5.64 SCF/lb VS) added which was higher than those of other CFCSTR two-phase runs. This methane yield was 70% of the theoretical methane yield. The upflow system gas yield was 87% of the ADP yield indicating nearly complete conversion of the biodegradable VS. As indicated in Table 93, the methane yield of the CFCSTR two-phase process was 37% higher than that of

TABLE 92. STEADY-STATE OPERATING CONDITIONS FOR ADVANCED UPFLOW TWO-PHASE DIGESTION RUNS CONDUCTED WITH HANOVER PARK SLUDGE

	Run no.	Digester no.	Mean temperature, °C	Mean HRT, days	Mean loading, kg VS/m^3-day
Upflow acid phase					
Mesophilic	UAP2MR*	338	35.4 (3)†	2.0 (12)	26.1 (13)
Mesophilic	UAP2M	338	36.0 (3)	2.3 (19)	21.4 (19)
Upflow methane phase					
Mesophilic	UMP5M	339	33.8 (3)	5.5 (11)	9.35 (11)
Meso-meso two-phase					
System (UAP2MR and UMP5M)	UTP7M-M	--	--	7.5 (11)	6.87 (11)

	Feed total solids concentration, g/L	Feed volatile solids concentration, g/L	Total run duration, days	Steady-state run duration, days
Upflow acid phase				
Mesophilic	67.6	51.3	73	37
Mesophilic	68.4	49.9	60	41
Upflow methane phase				
Mesophilic	--	--	73	37
Meso-meso two-phase				
System (UAP2MR and UMP5M)	67.6	51.3	73	37

[*] Methane-phase effluent from acid-phase run UAP2MR was recycled to this acid-phase digester at a rate of about 35 vol % of the system feed rate.

[†] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of standard deviation to the mean.

TABLE 93. COMPARISON OF STEADY-STATE GAS PRODUCTIONS FROM MESO-MESO UPFLOW TWO-PHASE, MESO-MESO CFCSTR TWO-PHASE AND MESOPHILIC CFCSTR SINGLE-STAGE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 7-DAY HRT[*]

	Upflow two-phase[†]			CFCSTR two-phase			
	Acid digester	Methane digester	System	Acid digester	Methane digester	System	CFCSTR single-stage
Run number	UAP2MR	UMP5M	UAP7M-M	----------	TP7M-M	----------	SSS7M
Operation							
Feed VS concentration, mg/L[+]	51,320	--	--	51,000	--	--	52,220
HRT, days	2.0	5.5	7.5	1.9	4.9	6.8	7.0
Loading, kg VS/m^3-day	26.13	9.35	6.87	26.38	10.08	7.29	7.51
Performance							
Total gas yield, SCM/kg VS added	0.144 (24)[**]	0.400 (12)	0.544 (16)	0.157 (16)	0.310 (11)	0.467 (12)	0.318 (13)
Methane yield, SCM/kg VS added	0.083 (25)	0.269 (12)	0.352 (15)	0.091 (16)	0.212 (12)	0.302 (13)	0.220 (16)
Gas composition, mol %							
Hydrogen	0.4	0.0	--	0.0	--	--	--
Methane	57.7	67.4	64.8	57.1	68.2	64.7	69.1
Carbon dioxide	41.2	32.2	34.6	42.5	31.6	35.1	30.6
Nitrogen	0.7	0.4	0.5	0.4	0.2	0.2	0.3
Total gas production rate, SCM/m^3-day	3.256 (16)	3.359 (10)	3.332 (12)	4.063 (5)	3.089 (5)	3.358 (4)	2.327 (12)
Methane production rate, SCM/m^3-day	1.874 (17)	2.263 (10)	2.160 (12)	2.343 (5)	2.100 (5)	2.173 (4)	1.609 (13)

[*] Data reported are means of all data collected during the steady-state period.

[†] In this upflow mode of operation acid-phase underflow was 12 vol % of the system flow rate. Also, methane digester effluents were recycled to the acid digester at the rate of 35 vol % of the system flow rate.

[+] Feed VS concentrations are weighted averages of the various feed slurry concentrations.

[**] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of standard deviation to the mean.

TABLE 94. COMPARISON OF STEADY-STATE EFFLUENT QUALITIES OF MESO-MESO UPFLOW TWO-PHASE, MESO-MESO CFCSTR TWO-PHASE, AND MESOPHILIC SINGLE-STAGE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 7-DAY HRT[*]

	Upflow two-phase[†]			CFCSTR Two-phase		
	Acid digester overflow	Acid digester underflow	Methane digester[+]	Acid digester	Methane digester[+]	CFCSTR single-stage
Run number	------ UAP2MP	------	UMP5M	----- TP7M-M	------	SS7M
HRT, days	------- 2.0	--------	5.5	1.9	4.9	7.0
Effluent, pH	6.77	6.66	7.29	6.63	7.30	7.06
Alkalinities, mg/L as $CaCO_3$						
Total	8325	8480	9415	7475	8100	6368
Bicarbonate	7020	5384	9248	6368	8068	6196
Volatile acids, mg/L						
Acetic	1277	2438	145	721	63	164
Propionic	1147	1518	180	728	29	104
Iso-butyric	266	425	0	109	0	0
Butyric	377	1024	0	160	0	0
Iso-valeric	581	688	0	136	0	0
Valeric	192	361	3	61	10	0
Caproic	37	31	4	31	33	0
Total as acetic	3118 (16)[**]	5289 (18)	295 (51)	1627 (15)	109 (44)	248 (29)
Ethanol, mg/L	4	18	0	0	0	3
Nitrogen, mg/L						
Ammonia-N	1269	1185	1378	918	1049	728
Organic-N	2000	2285	1891	2241	1845	1966
Solids, mg/L						
TS	60,150	70,260	59,640	60,720	50,690	64,350
VS	42,980	51,920	39,790	42,480	32,810	39,585
Organic components, mg/L						
Crude protein	12,500	14,281	11,819	14,006	11,531	10,427
Carbohydrates			5650	7791	4932	7014
Lipids	9259	11,661	4849	7178	3824	11,389

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[*] Data reported are means of one or more determinations made during the steady-state period.

[†] In this upflow mode of operation acid-phase underflow was 12 vol % of the system flow rate. Also, methane digester effluents were recycled to the acid digester at the rate of 35 vol % of the system flow rate.

[+] Numbers in parentheses are coefficients of variation, expressed as the percent ratio of standard deviation to the mean.

[**] System effluent qualities are the same as those of the methane digester.

single-stage CFCSTR digestion under similar operating conditions showing the beneficial effect of the phase-separated fermentation mode. Similarly, the methane yield of the upflow two-phase process was about 17% higher than that of CFCSTR two-phase high-rate digestion. This additional increase in methane yield could be attributed to the reactor effect. Other notable performance characteristics of the upflow two-phase process were as follows:

- The upflow acid digester gases contained hydrogen gas and a higher concentration of nitrogen gas than the CFCSTR acid digester. The presence of hydrogen gas in the upflow acid digester indicated that there was no acetogenic activity in this reactor. Hydrolysis and acidification were the major reactions in the upflow acid digester.

- Volatile acids production in the upflow acid digester was much higher than that in the CFCSTR acid digester (Table 95); acids concentrations in the underflow were about double those in the overflow. Increased acids production in the upflow acid digester was due to the fact that this reactor had an SRT which was considerably higher than that of the CFCSTR digester.

- The upflow acid and methane digesters developed bicarbonate alkalinities which were considerably higher than those of the CFCSTR digesters, and therefore was less prone to upsets owing to organic overloads.

- The upflow two-phase digestion process effected much higher protein, carbohydrate, and lipid reduction than the CFCSTR two-phase digestion process (Table 96).

Data presented in Table 95 showed that the right (or second) chamber of the acid digester accumulated C_3 and higher volatile acids in larger concentrations than the first chamber. This observation suggests that the hydrolysis and liquefaction processes continued to be operative in the second chamber indicating effective utilization of the entire digester.

Volatile acids concentration profile in the upflow methane digester showed that acetate as well as the higher acids were readily converted to their respective end-products within the bottom one-half of the culture depth. Acetogenesis, aceticlastic methane formation, and syntrophic methane fermentation were the predominant reactions in the upflow methane digester.

Effect of Inter-Phase Effluent Recycling

The steady-state upflow two-phase run discussed above was conducted with methane-phase effluents recycled to the feed side of the acid-phase digester. The intent of the recycle was to promote removal of hydrogen (or electron flow) and substrate oxidation to produce volatile acids or their precursors. Hydrogen is removed by reduction of sulfates, nitrates/nitrites, and carbon dioxide with the production of sulfides, and nitrogen and methane gases by the sulfate-reducing, denitrifying, and hydrogen-utilizing syntrophic methane bacteria. Steady-state data reported in Table 97 show that for similar operating conditions, methane and nitrogen yields and production rates in the acid digester were much higher when methane digester effluents were recycled

TABLE 95. STEADY-STATE pH, ORP, AND VOLATILE ACIDS AND ETHANOL CONCENTRATION PROFILES IN MESO-MESO UPFLOW TWO-PHASE DIGESTERS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 7-DAY HRT*

	Left chamber (influent side)	Underflow (right chamber on effluent side)	Overflow	Bottom port	11.5-L port	15.5-L port	Effluent (19-L port)
Run number	UAP2MR			UMP5M			
pH	--	6.66	6.77	--	7.29	--	7.29
ORP, mV	--	-234	-241	--	-327	--	-371
Volatile acids, mg/L							
Acetic	2576	2438	1277	3	173	211	145
Propionic	1282	1518	1147	0	290	311	180
Iso-butyric	314	425	266	0	0	0	0
Butyric	990	1024	377	0	0	0	0
Iso-valeric	368	688	581	0	0	0	0
Valeric	218	361	192	0	3	3	3
Caproic	39	31	37	0	4	4	4
Total as acetic	4870	5289	3118	3	412	467	295
Ethanol, mg/L	89	18	4	0	0	0	0

* Data reported are means of all determinations made during the steady-state period.

TABLE 96. COMPARISON OF STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF MESO-MESO UPFLOW TWO-PHASE, MESO-MESO CFCSTR TWO-PHASE, AND MESOPHILIC SINGLE-STAGE SYSTEMS OPERATED WITH HANOVER PARK SLUDGE AT ABOUT A 7-DAY HRT[a]

	Upflow two-phase[b]			CFCSTR two-phase			
	Acid digester	Methane digester	System	Acid digester	Methane digester	System	CFCSTR single-stage
Run number	UAP2MR	UMP5M	UTP7M-M	----------	TP7M-M	----------	SS7M
HRT, Days	2.0	5.5	7.5	1.9	4.9	6.8	7.0
VS reduction, %							
MOP_{16}[c]	14.1	21.2	32.3	15.7	21.2	33.6	18.3
Wt-of-gas basis[d]	14.6	37.1	51.7	18.1	33.4	51.5	32.7
Carbon-in-gas basis[e]	11.8	33.1	44.9	14.3	28.5	42.8	28.8
Based on the theoretical gas yield[f]	13.4	37.1	50.5	14.6	28.8	43.3	29.5
Biodegradable VS reduction[g]	23.0	64.0	87.0	25.1	49.6	74.7	50.9
Reduction of organic components, %							
Crude protein	29.0	7.0	34.0	9.6	17.7	25.6	26.2
Carbohydrates	29.0	36.4	54.8	29.1	36.6	55.1	26.4
Lipids	37.8	49.1	68.4	28.2	46.7	61.8	40.0
ΣCPL[h]	32.0	28.3	51.2	20.6	30.0	44.4	32.4

[a] Data reported are means of one or more determinations made during the steady-state period.

[b] Methane phase effluents were recycled to the acid-phase digester at a rate of 35 vol % of the system feed rate.

[c] These VS reductions were calculated according to the following formula: $VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[d] These VS reductions were calculated according to the following formula: $VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[e] These VS reduction were calculated according to the following formulas: $VS_R = 100 \times (1.84 \times$ mass flow rate of product gas carbon)/mass flow rate of VS fed.

[f] These VS reductions were calculated by expressing the observed total gas yield as a percentage of the theoretical gas yield of 1.078 SCM/kg VS added.

[g] The biodegradable VS reductions were calculated by dividing the theoretical-gas-yield-based VS reduction by a biodegradability factor of 0.58.

[h] ΣCPL means the sum of the masses of carbohydrates, crude protein, and lipids.

TABLE 97. COMPARISON OF STEADY-STATE GAS PRODUCTIONS FROM MESOPHILIC UPFLOW ACID-PHASE DIGESTION OF HANOVER PARK SLUDGE WITH AND WITHOUT METHANE-PHASE EFFLUENT RECYCLE

	Mesophilic without recycle	Mesophilic with recycle*
Run number	UAP2M	UAP2MR
Operation		
Feed VS concentration, mg/L†	49,900	51,320
HRT, days	2.3	2.0
Loading, kg VS/m^3-day	21.36	26.13
Performance		
Total yield, SCM/kg VS added	0.090	0.144
Methane yield, SCM/kg VS added	0.052	0.083
Gas composition, mol %		
Hydrogen	--	0.4
Methane	57.7	57.7
Carbon dioxide	41.8	41.2
Nitrogen	0.5	0.7
Total gas production rate, SCM/m^3-day	1.928	3.256
Methane production rate, SCM/m^3-day	1.112	1.874
Nitrogen production rate, SCM/m^3-day	0.0096	0.0228

* Methane phase effluent was recycled to the acid-phase digester at 35% of the system feed rate.

† Feed VS concentration are weighted averages of the various feed slurry concentrations.

to it than those observed without such recycle. Data presented in Table 98 show that the rates of volatile acids production with methane-phase effluent recycle were higher than those obtained without recycle. These observations strongly suggested that methane-digester effluent recycling accelerated hydrolysis and acidification of the sludge solids.

Thermophilic Two-Phase Digestion With Innovative Upflow Reactors

Thermophilic Acid-Phase Digestion--

Upon termination of the mesophilic runs, the upflow acid-phase digester was acclimated to a thermophilic temperature; the overflow effluents from the acid digester were fed to the upflow thermophilic methane digester. Methane digester effluents were not recycled to the acid digester, as it was during the mesophilic operation. Operating data from three consecutive acid-phase runs at a 2-day HRT are presented in Tables 99 and 100 and show that gas and methane productions and gas-phase methane contents under thermophilic conditions were much lower than those under mesophilic conditions. Examination of the volatile acids production data for acid-phase Runs UAP2T, UAP2.1T and UAP1.9T shows that although thermophilic acids production during the initial Run UAP2T was higher than that at the mesophilic temperature, the volatile acid yields dropped upon continued operation at the 2-day HRT (Table 100).

Meso-Thermo and Thermo-Thermo Upflow Two-Phase Digestion

In the meso-thermo two-phase run, the upflow acid digester had a mesophilic temperature of 35°C while the upflow methane digester was maintained at a temperature of 55°C. The HRT's of the acid and methane digesters were 4.5 and 12.1 days, respectively. Operating and performance data presented in Tables 101 and 102 show that the mesophilic acid-phase digester exhibited higher gas and methane yield and production rates and methane content than the methane digester. Concentrations of all individual volatile acids were about 50% higher in the thermophilic methane digester compared to those in the mesophilic acid digester despite the fact that the HRT of the former digester was three times that of the latter. Data presented in Table 103 showed that additional volatile acids over those prevalent in the acid digester were produced in the methane digester. It was obvious from these observations that the thermophilic methane digester experienced little acetogenic and methanogenic conversions, and that it behaved as an acid digester. The meso-thermo upflow two-phase operation was discontinued after about two weeks of operation.

In the next upflow two-phase run the acid digester temperature was changed to 55°C for thermophilic operation. It was rationalized that if the acid digester was maintained at a thermophilic temperature then the liquefaction-acidification process would be enhanced in this digester with a concommitant decline in acidification activity in the thermophilic methane digester.

In the thermo-thermo two-phase run the acid and the methane digesters were operated at HRT's of about 2.1 and 5.4 days, respectively. The perform-

TABLE 98. COMPARISON OF VOLATILE ACIDS PRODUCTION RATES FROM MESOPHILIC AND THERMOPHILIC UPFLOW ACID-PHASE DIGESTION OF HANOVER PARK SLUDGE WITH AND WITHOUT METHANE-PHASE EFFLUENT RECYCLE

	Mesophilic without recycle, overflow	Mesophilic with recycle*
Run number	UAP2M	UAP2MR
Effluent pH	6.60	6.67
Volatile acids, g/day		
Acetic	4.14	4.96
Propionic	4.40	4.17
Iso-butyric	0.86	1.00
Butyric	1.25	1.59
Iso-valeric	1.18	2.08
Valeric	0.89	0.74
Caproic	0.28	0.13
Total as acetic	10.51	11.83
Total	13.00	14.66
Ethanol, g/day	0.00	0.02

* Methane-phase effluent was recycled to the acid-phase digester at a rate of 35% of the system feed rate. Effluent from this run was continuously wasted from the top (overflow) and bottom (underflow) of the digester. The overflow and underflow rates were 88 and 12% of the total effluent rate, respectively.

TABLE 99. COMPARISON OF GAS PRODUCTIONS FROM MESOPHILIC AND THERMOPHILIC UPFLOW ACID-PHASE DIGESTION OF HANOVER PARK SLUDGE

	Thermophilic (55°C)	Thermophilic (54°C)	Thermophilic (54°C)	Mesophilic (35°C)
Run number	UAP2T	UAP2.1T	UAP1.9T	UAP2M
Run duration, days	16	9	7	41
Operation				
Feed VS concentration, mg/L*	50,200	49,930	24,900	49,900
HRT, days	2.0	2.1	1.9	2.3
Loading, kg VS/m^3-day	24.55	24.24	13.11	21.36
Performance				
Total yield, SCM/kg VS added	0.021	0.020	0.030	0.090
Methane yield, SCM/kg VS added	0.009	0.009	0.014	0.052
Gas composition, mol %				
Hydrogen	--	0.0	--	--
Methane	44.8	45.8	46.4	57.7
Carbon dioxide	52.6	48.5	49.6	41.8
Nitrogen	2.6	5.7	4.0	0.5
Total gas production rate, SCM/m^3-day	0.525	0.475	0.394	1.928
Methane production rate, SCM/m^3-day	0.235	0.218	0.183	1.112

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* Feed VS concentration are weighted averages of the various feed slurry concentrations.

TABLE 100. COMPARISON OF EFFLUENT QUALITIES FROM MESOPHILIC AND THERMOPHILIC UPFLOW ACID-PHASE DIGESTION OF HANOVER PARK SLUDGE WITHOUT METHANE-PHASE EFFLUENT RECYCLE

	Thermophilic (55°C)	Thermophilic (54°C)	Thermophilic (54°C)	Mesophilic (35°C)
Run number	UAP2T	UAP2.1T	UAP1.9T	UAP2M
Effluent, pH	6.44	6.48	6.55	6.60
Volatile acids, mg/L				
Acetic	2516	1360	803	1183
Propionic	1531	693	414	1256
Iso-butyric	514	226	153	246
Butyric	792	411	188	358
Iso-valeric	972	452	268	337
Valeric	54	120	8	254
Caproic	71	74	46	79
Total as acetic	5287	2731	1557	3002
Ethanol, mg/L	10	16	0	0

TABLE 101. COMPARISON OF GAS PRODUCTIONS FROM MESO-THERMO AND THERMO-THERMO UPFLOW TWO-PHASE AND THERMO-THERMO-THERMO THREE-STAGE DIGESTION SYSTEMS OPERATED WITH HANOVER PARK SLUDGE

	Meso-thermo two-phase			Thermo-thermo two-phase		
	Mesophilic (35°C) acid digester	Thermophilic (55°C) methane digester	Meso-thermo two-phase system	Thermophilic (54°C) acid digester	Thermophilic (52°C) methane digester	Thermo-thermo system
Run number	UAP4.5M	UMP12T	UTP17M-T	UAP2.1T	UMP5.4T	UTP7T-T
Operation						
Feed VS concentration, mg/L*	49,100			49,600		
HRT, days	4.46	12.09	16.55	2.06	5.35	7.41
Loading, kg VS/m^3-day	11.22	4.13	3.02	24.24	9.34	6.74
Performance						
Total gas yield, SCM/kg VS added	0.182	0.060	0.242	0.020	0.029	0.049
Methane yield, SCM/kg VS added	0.109	0.032	0.141	0.009	0.015	0.024
Gas composition, mol %						
Hydrogen	--	--	--	0.0	--	--
Methane	59.8	53.8	58.3	45.8	52.0	49.4
Carbon dioxide	39.7	45.2	41.1	48.5	45.6	47.0
Nitrogen	0.5	1.0	0.6	5.7	2.4	3.6
Total gas production rate, SCM/m^3-day	2.046	0.248	0.732	0.475	0.271	0.326
Methane production rate, SCM/m^3-day	1.224	0.133	0.426	0.218	0.141	0.160

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* Feed VS concentrations are weighted averages of the various feed slurry concentrations.

(continued)

TABLE 101 (continued)

	Thermo-thermo-thermo three-stage			
	Thermophilic (54°C) acid digester	Thermophilic (49°C) upflow methane digester	Thermophilic (52°C) CFCSTR methane digester	Three-stage system
Run number	UAP1.9T	UMP5.2T	CMP13T	UTP20T-T-T
Operation				
Feed VS concentration, mg/L*	24,900			
HRT, days	1.91	5.18	13.30	20.39
Loading, kg VS/m^3-day	13.11	4.83	1.88	1.23
Performance				
Total gas yield, SCM/kg VS added	0.030	0.051	0.267	0.348
Methane yield, SCM/kg VS added	0.014	0.031	0.209	0.254
Gas composition, mol %				
Hydrogen	—	—	—	—
Methane	46.4	61.1	78.3	72.9
Carbon dioxide	49.6	38.3	20.8	25.9
Nitrogen	4.0	0.6	0.9	1.2
Total gas production rate, SCM/m^3-day	0.394	0.249	0.501	0.427
Methane production rate, SCM/m^3-day	0.183	0.152	0.392	0.311

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* Feed VS concentrations are weighted averages of the various feed slurry concentrations.

TABLE 102. COMPARISON OF EFFLUENT QUALITIES FROM MESO-THERMO AND THERMO-THERMO UPFLOW TWO-PHASE AND THERMO-THERMO-THERMO THREE-STAGE DIGESTION SYSTEMS OPERATED WITH HANOVER PARK SLUDGE

	Meso-thermo two-phase		Thermo-thermo two-phase		Thermo-thermo-thermo three-stage		
	Mesophilic (35°C) acid digester	Thermophilic (55°C) methane digester*	Thermophilic (54°C) acid digester	Thermophilic (52°C) methane digester*	Thermophilic (54°C) upflow acid digester	Thermophilic (49°C) upflow methane digester	Thermophilic (52°C) CFCSTR methane digester*
Run number	UAP4.5M	UMP12T	UAP2.1T	UMP5.4T	UAP1.9T	UMP5.2T	CMP13T
Effluent, pH	6.90	7.20	6.48	7.06	6.55	6.78	7.64
Volatile acids, mg/L							
Acetic	1046	1661	1360	1742	803	1345	229
Propionic	976	1465	693	1264	414	685	782
Iso-butyric	116	368	226	339	153	235	178
Butyric	162	207	411	437	188	227	0
Iso-valeric	224	678	452	628	268	410	303
Valeric	114	0	120	158	8	43	13
Caproic	104	112	74	2	46	73	78
Total as acetic	2280	3697	2731	3759	1557	2519	1211
Ethanol, mg/L	0	0	16	18	0	0	0

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* System effluent quantities are the same as those of the methane phase digester.

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TABLE 103. SOLIDS, pH, ORP, AND VOLATILE ACIDS CONCENTRATION PROFILES IN THERMOPHILIC UPFLOW METHANE-PHASE DIGESTER OPERATED IN TANDEM WITH THERMOPHILIC UPFLOW ACID-PHASE DIGESTER

	Acid-phase effluent	Methane-phase bottom port	Methane-phase 7-L port	Methane-phase 11.5-L port	Methane-phase 15.5-L port	Methane-phase 19-L port (top)	Methane-phase overflow (effluent)
Run number	UAP2.1T	UMP5.4T					
Solids, g/L							
Total	--	68.56	78.16	66.83	62.86	64.00	57.70
Volatile	--	47.02	47.59	44.84	42.69	43.75	39.94
Fixed	--	21.54	30.57	21.99	20.17	20.25	17.76
pH	6.48	6.98	7.02	7.04	6.99	7.03	7.06
ORP, mV	-371	-399	-391	-378	-360	-317	-373
Volatile acids, mg/L							
Acetic	1360	1643	1732	1708	1769	1720	1742
Propionic	693	1153	1248	1248	1264	1263	1264
Iso-butyric	226	320	339	339	347	340	339
Butyric	411	443	462	462	477	467	437
Iso-valeric	452	601	642	642	658	643	628
Valeric	120	166	155	152	155	157	158
Caproic	74	4	6	6	6	7	2
Total as acetic	2731	3551	3762	3736	3836	3769	3759
Ethanol, mg/L	16	0	0	0	0	0	0

ance of the upflow acid digester changed dramatically when the operating temperature was changed from mesophilic to thermophilic. As would be evident from Tables 101 and 102, gas and methane production, and gas-phase methane content decreased, and volatile acids production and denitrification activity increased substantially in the acid digester as a result of this temperature change. Gas and methane yields from the upflow thermophilic methane digester were lower when it received thermophilic acid digester effluent than they were when it was fed with mesophilic acid digester effluents. Surprisingly, volatile acids accumulations in the thermophilic methane digester at an HRT of 5.4 days during the thermo-thermo run were about the same as those experienced at an HRT of 12 days during the meso-thermo run. Clearly, conversion of the upflow acid digester from mesophilic to thermophilic operation, and decrease in methane digester HRT from 12 to 7 days had no effect on the performance of the methane digester. As would be evident from Table 101, methane yield from the thermo-thermo upflow two-phase process at a system HRT of about 7 days was about one-seventh that of the meso-thermo two-phase run at an HRT of about 17 days. However, methane yields from both the above two-phase runs were very low. The thermo-thermo two-phase run was terminated after about 9 days of operation.

The results of the meso-thermo and thermo-thermo upflow two-phase runs demonstrated that the thermophilic upflow methane digester showed little acetogenic and methanogenic activities. In addition, gas production from the upflow acid digester decreased substantially when the operating temperature was changed from mesophilic to thermophilic. Thus, the inhibitory effects of thermophilic temperature observed during CFCSTR single-stage and two-phase runs were also observed during upflow two-phase operation; in fact, the inhibitory effects of the thermophilic metabolites was more severe on the upflow thermophilic digesters than the CFCSTR thermophilic digesters. This is evidenced by the fact that methane yield and production rate from the upflow thermophilic methane digester were considerably lower than those of the CFCSTR thermophilic methane digester (see Tables 93 and 101).

THERMO-THERMO-THERMO UPFLOW THREE-STAGE DIGESTION

In the three-stage thermo-thermo-thermo operation, the upflow acid and the upflow methane digesters were operated at HRT's of about two and 5.2 days as in the case of the thermo-thermo two-phase run discussed above (Table 101). In addition, a CFCSTR methane-phase digester was also operated at an HRT of about 13.3 days and in series with the thermo-thermo upflow two-phase system to promote gasification of the accumulated volatile acids. The CFCSTR thermophilic methane digester was first operated at about 60°C — this thermophilic temperature is higher than the "normal" thermophilic temperature of 55°C — expecting that methanogenic conversion at this temperature would be better than at the 55°C temperature used for all thermophilic operations. It was observed that gas production at 60°±1°C was very low (about 1 L/day) indicating that this digester temperature was unacceptable for CFCSTR methane-phase operation. A thermophilic temperature of 52°±1°C was used next. In response to this change, gas production from the thermophilic digester increased from 1 L/day at 60°C to 20 L/day at 52°C showing that the latter temperature should be preferred. The three-stage system was operated at about one-half the loading rate of the meso-meso and the meso-thermo two-phase systems to alleviate inhibition of the gasification process during thermophilic operation. The operating and performance data for the three-stage system are presented in Tables 101 and 102. It is apparent from these data that the three-stage system performed better than the meso-thermo or the thermo-thermo two-phase runs primarily because the CFCSTR thermophilic methane digester exhibited higher gasification efficiency than the upflow thermophilic methane digester.

Information compiled from the thermo-thermo upflow two-phase digestion runs seemed to indicate that a much higher degree of inhibition of thermophilic digestion was experienced in the high-SRT upflow digester than in the CFCSTR digester. This may be due to the fact that whereas there is continual flushing of digesting substrate solids and their breakdown products in a CFCSTR digester, these substances, which plausibly produce inhibitors for the thermophiles, accumulated in the upflow digesters; thus, unlike the CFCSTR digesters, the upflow reactors contained a larger reservoir of the inhibitor-producing compounds. This is apparent considering that for similar dilution-rate and loading-rate conditions, the thermophilic upflow methane digester contained 40-47 g/L of VS and 3600-3800 mg/L of volatile acids (see solids and VA profiles in Table 103) compared with 34-g/L of VS and about 2100 mg/L of VA in the thermophilic CFCSTR single-stage system (see Table 51, Section 11). The three-stage thermophilic run was terminated after about 10 days of operation (in October 1984).

FINAL THERMO-THERMO UPFLOW TWO-PHASE RUN

Based on the experience gained from the thermophilic upflow two-phase digestion runs described above, it was decided that successful thermophilic operation of the upflow two-phase system could be achieved perhaps with prolonged enrichment and acclimation of the acidogenic and methanogenic populations under conditions of gradually increasing loading and hydraulic dilution rates and by changing the digester feed. The upflow acid and methane digesters were operated according to this strategy for about two months with Hanover Park sludge; the operating and performance data for the last three weeks of this run, operated with mixed Downers Grove primary and Stickney activated sludges are depicted in Figures 18 through 20. These figures show that with gradual acclimation of the acidogenic and methanogenic thermophiles to decreasing HRT and increasing loading rate, it was possible to steadily improve the methane yields and production rates of the upflow two-phase system. At the time of termination of this run, the methane yield and production rate, and system effluent volatile acids concentration of this thermo-thermo upflow two-phase system were 0.32 SCM/kg VS added, 1.40 vol/vol-day, and 2100 mg/L, respectively, which compared well with the corresponding performance parameters of the thermophilic CFCSTR two-phase process (see Tables 61 and 62, Section 12).

Although operation of the above upflow thermophilic system could not be continued due to time constraints, data collected during the transient phase of operation suggested that upflow thermophilic digesters could be sensitive to certain feed sludges and that a long enrichment and acclimation period is required before efficient system operation can be expected.

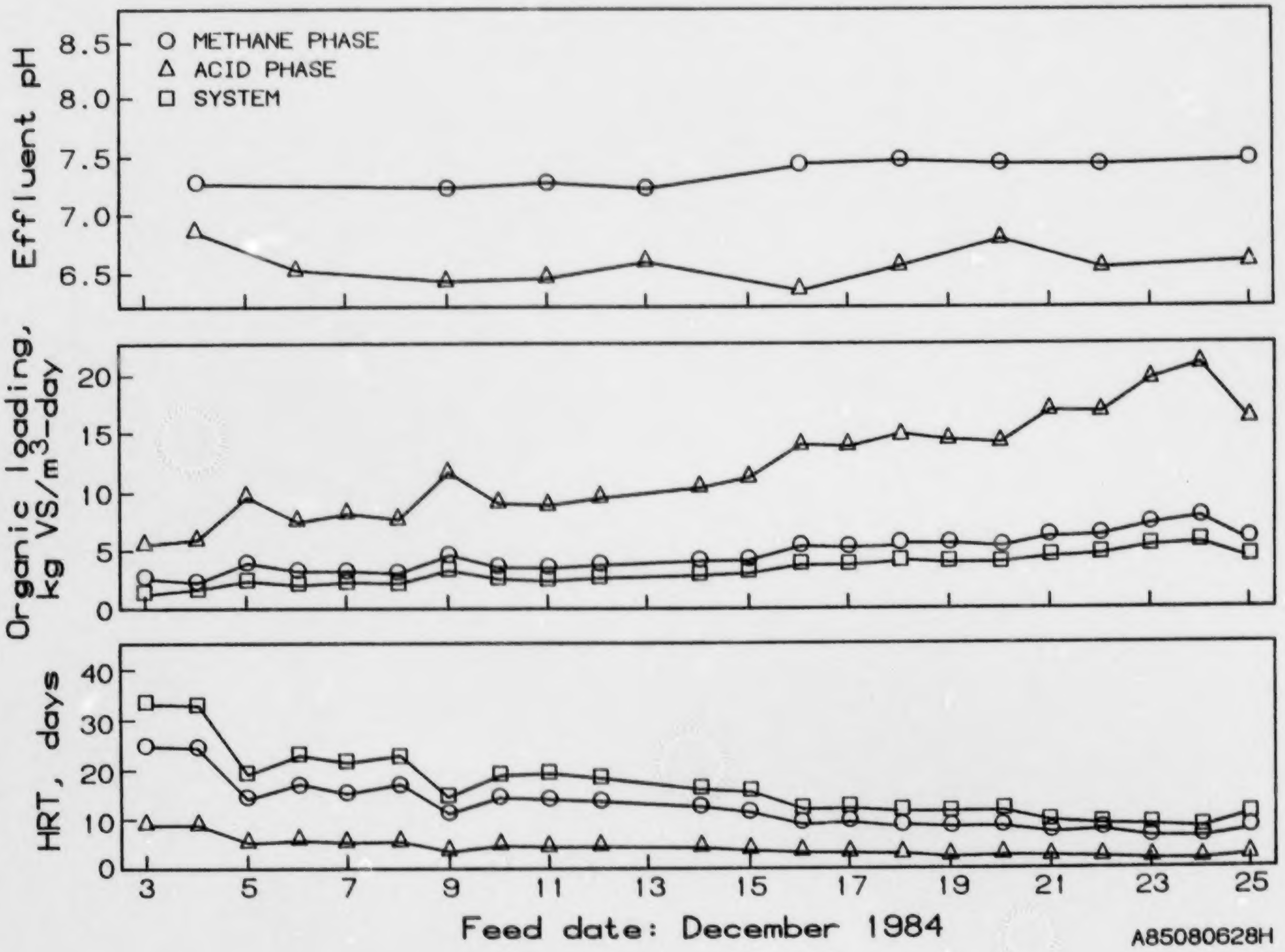

Figure 18. Operating conditions of the thermo-thermo two-phase system fed with a mixture of Downers Grove primary and Stickney activated sludge.

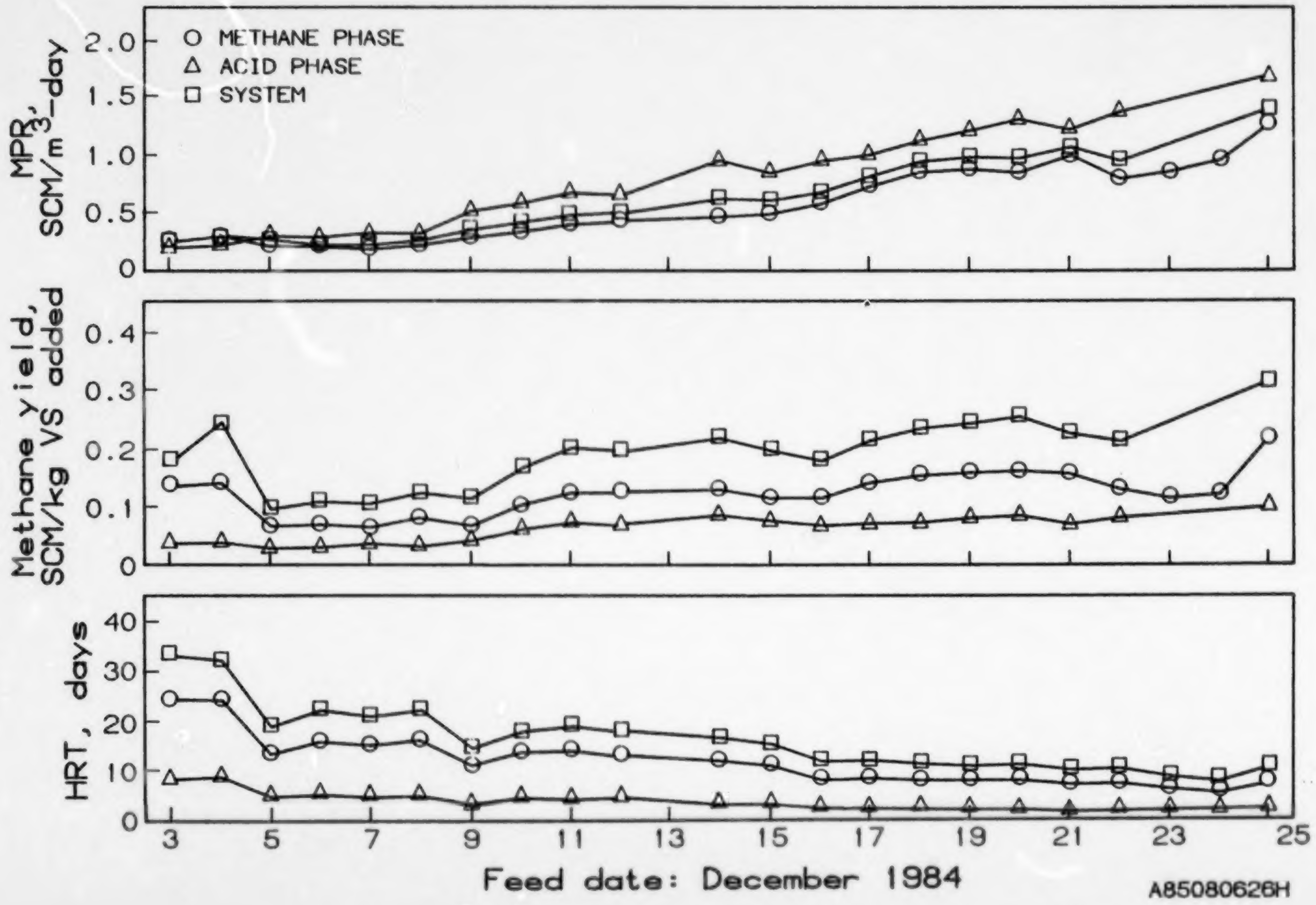

Figure 19. Methane yield and production rate from the thermo-thermo two-phase system fed with a mixture of Downers Grover primary and Stickney activated sludges.

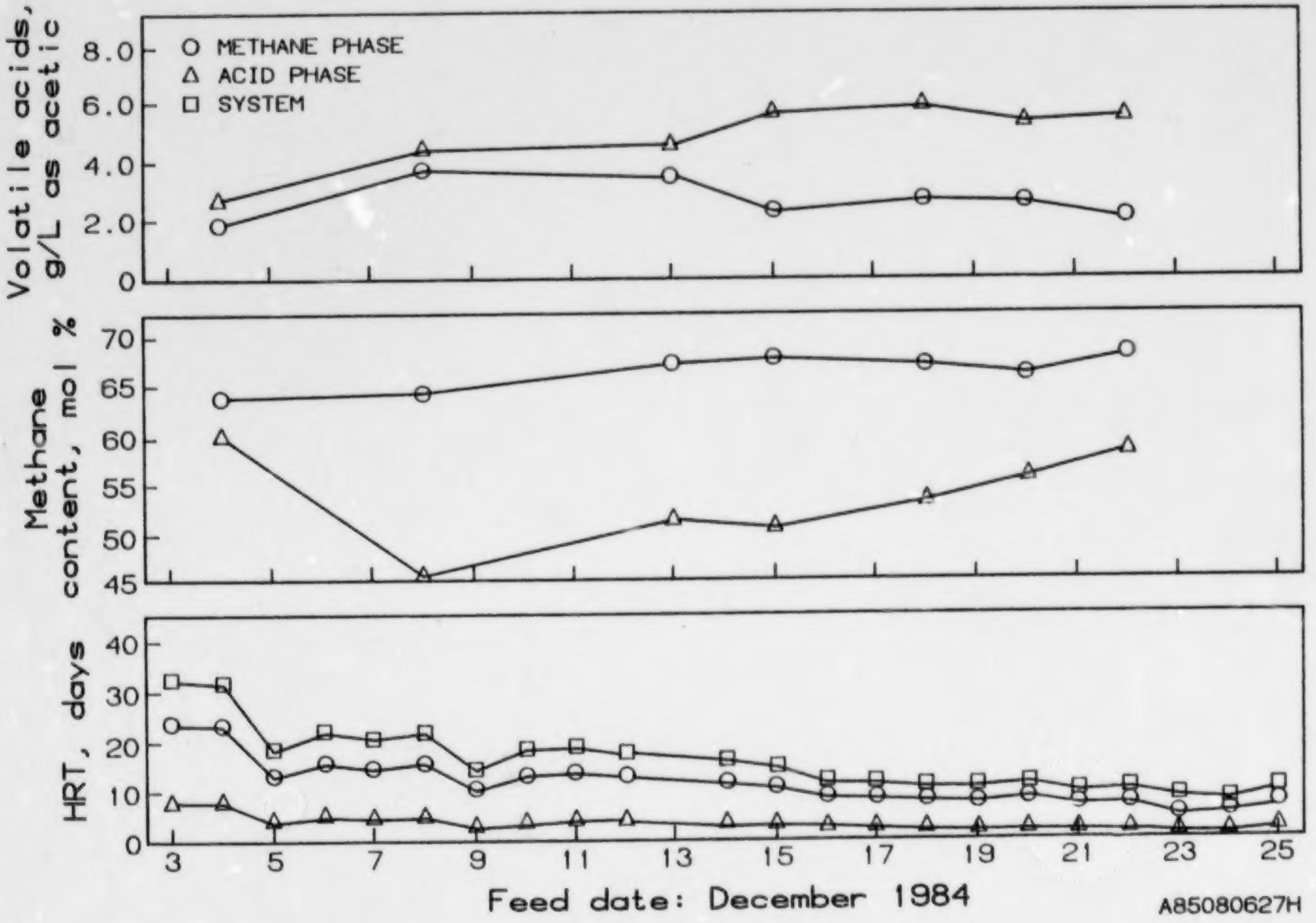

Figure 20. Methane content and effluent volatile acids of the thermo-thermo two-phase system fed with a mixture of Downers Grove primary and Stickney activated sludges.

TWO-PHASE PROCESS IMPROVEMENT WITH ENZYME TREATMENT OF DIGESTER FEED

As described in Section 6, efforts were directed towards improving two-phase process performance by pretreating the feed sludge with cellulase-cellobiase while also dosing the acid-digester with lipase. These enzymes were expected to accelerate hydrolysis of the polymeric cellulosic and lipid particles, thereby promoting further conversion of the hydrolytic products to volatile fatty acids, hydrogen, and carbon dioxide which are substrates for methane fermentation.

That the selected cellulase-cellobiase enzyme system was an effective hydrolyzer is evident from the data in Table 104 which shows that during incubation (and before digestion) the enzyme-treated feed produced more than twice as much volatile acids as produced by the untreated raw sludge. The presence of residual hydrogen gas in this pretreatment vessel was indicative of the occurrence of oxidation reactions involved in the conversion of sludge hydrolysates to fatty acids. Since methane and nitrogen are end products of hydrogenation of carbon dioxide, and nitrates, respectively, and since the contents of these gases in the pre-treatment vessel were unusually high and that of carbon dioxide was very low, if could be concluded that considerable dehydrogenation of the sludge feed occurred following cellulose-cellobiase treatment and prior to the addition of this sludge to the digester.

Comparison of two-phase digestion data obtained at a system HRT of three days with untreated and enzyme-treated sludges showed that gas and methane yields and production rates from the mesophilic acid and methane digesters and the two-phase system receiving enzyme treatment were considerably higher than those observed with the untreated sludge feed (Tables 105 and 106). The methane contents of the digesters' gases were also higher when enzyme treatment was used. As expected, the acid-phase and the two-phase system carbohydrate reductions of about 50% and 64% obtained with cellulase-cellobiase treatment of the feed were much higher than those observed with untreated feeds (Table 107). Similarly, methane-phase and two-phase system lipid reductions with lipase addition were 36% and 39% compared with about 9% and 27%, respectively observed without such treatment. Gas and methane yields and production rates from digestion of the enzyme treated sludge were significantly higher than those from digestion of untreated sludge (Table 105). Similarly, residual effluent volatile acids from the system receiving enzyme-treated feed were much lower than those from the two-phase system receiving untreated sludge (Table 106).

It is noteworthy that lipase dosing had the effect of shifting lipid reduction from the acid to the methane digester, probably because this external enzyme was relatively ineffective at the low pH of 6.4 prevalent in the former digester. Another reason for this low lipid reduction in the acid digester could be that with cellulase-cellobiase treatment, the acidogenic organisms metabolized carbohydrates in preference to the more recalcitrant lipid substrates because this mode of fermentation was energetically more favorable. Lipase activity in the acid digester receiving enzyme-treated feed appeared to be much lower than that evidenced with untreated feeds. The acid digester exhibited higher indigenous lipolytic activity than the methane digester during two-phase process operation with untreated raw sludge.

TABLE 104. EFFECT OF CELLULASE-CELLOBIASE PRETREATMENT ON VOLATILE ACIDS AND GAS PRODUCTION DURING INCUBATION FROM MIXED DOWNERS GROVE PRIMARY AND STICKNEY ACTIVATED DIGESTER FEED SLUDGES

	Untreated feed slurry	Enzymatically pretreated feed slurry*
pH		
Initial	6.25	4.69
After 24 hrs	--	5.77
Volatile acids, mg/L		
Acetic	1484	1891
Propionic	799	965
Iso-butyric	0	395
Butyric	214	1538
Iso-valeric	0	828
Valeric	42	918
Caproic	43	42
Total as acetic	2324	5739
Ethanol, mg/L	171	402
Gas composition, mol %		
Hydrogen	--	4.0
Methane	--	88.3
Carbon dioxide	--	6.4
Nitrogen	--	1.3

* This raw feed was pretreated for 24 hours at 35°C with cellulose (Novo Celluclast 1.5L) and cellobiase (Novozym 188) enzymes at dosages of 2.76 g/kg feed TS and 0.28 g/kg feed TS, respectively. Prior to pretreatment the feed pH was adjusted to <5 with 2.5 N HCl.

TABLE 105. EFFECT OF CELLULASE-CELLOBIASE AND LIPASE TREATMENT ON STEADY-STATE GAS PRODUCTIONS FROM MESO-MESO CFCSTR TWO-PHASE SYSTEMS OPERATED WITH MIXED DOWNERS GROVE PRIMARY AND STICKNEY ACTIVATED SLUDGES AT AN HRT OF ABOUT 3 DAYS[*]

	Two-phase digestion with untreated raw sludge			Two-phase digestion with enzyme treated sludge[†]		
	Acid digester	Methane digester	System	Acid digester	Methane digester	System
Run number		TP3M-M			TP3M-M(E)	
Operation						
Feed Vs concentration, mg/L[+]		46,600			47,420	
HRT, days	0.91	2.15	3.06	0.93	2.31	3.24
Loading, kg VS/m^3-day	51.21	21.68	15.23	50.99	20.49	14.62
Performance						
Total gas yield, SCM/kg VS added	0.108 (7)[**]	0.197 (9)	0.305 (6)	0.121 (23)	0.230 (18)	0.351 (18)
Methane yield, SCM/kg VS added	0.057 (10)	0.124 (9)	0.180 (7)	0.070 (26)	0.152 (19)	0.222 (19)
Gas composition, mol %						
Hydrogen	0.05	0.0	0.0	0.05	--	--
Methane	52.0	63.1	59.1	57.3	65.9	63.0
Carbon dioxide	47.3	36.5	40.4	42.3	34.1	36.9
Nitrogen	0.7	0.4	0.5	0.4	0.0	0.1
Total gas production rate, SCM/m^3-day	5.575 (6)	4.293 (13)	4.674 (12)	6.245 (12)	4.749 (7)	5.178 (7)
Methane production rate, SCM/m^3-day	2.898 (9)	2.708 (14)	2.764 (12)	3.567 (15)	3.128 (7)	3.254 (7)

---

[*] Data reported are the means of all data collected during the steady-state period.

[†] The feed slurry for this run was pretreated with cellulase (Novo Celluclast 1.5 L) and cellobiase (Novozym 188) at dosages of 2.76 g/kg feed TS and 0.28 g/kg feed TS, respectively. Lipase (Novozym 225) was added to the acid digester at a dosage of 2.75 g/kg feed TS.

[+] Feed VS concentrations are weighted averages of the various feed slurry concentrations.

[**] Numbers in parentheses are the coefficients of variation expressed as the percent ratios of standard deviation to the mean.

TABLE 106. EFFECT OF CELLULASE-CELLOBIASE AND LIPASE TREATMENT ON STEADY-STATE EFFLUENT QUALITIES OF MESO-MESO CFCSTR TWO-PHASE SYSTEMS OPERATED WITH MIXED DOWNERS GROVE PRIMARY AND STICKNEY ACTIVATED SLUDGES AT ABOUT A 3-DAY HRT

	Two-phase digestion with untreated raw sludge		Two-phase digestion with enzyme treated sludge[b]	
	Acid digester	Methane digester[c]	Acid digester	Methane digester[c]
Run number	----- TP3M-M ------		---- TP3M-M(E) -----	
HRT, days	0.91	2.15	0.93	2.31
Effluent, pH	6.48	7.19	6.36	7.19
Volatile acids				
Acetic	2177	218	1206	208
Propionic	1403	1502	1310	978
Iso-butyric	288	50	230	27
Butyric	749	0	585	4
Iso-valeric	596	195	426	60
Valeric	1907	136	626	17
Caproic	51	31	33	12
Total as acetic	5518 (16)[d]	1680 (15)	3458 (28)	1066 (20)
Ethanol, mg/L	19	0	11	5
Solids, mg/L				
TS	61,600	57,450	60,410	54,900
VS	39,320	35,040	39,170	33,820
Organic compounds, mg/L				
Crude protein	12,044	10,512	--	--
Carbohydrates	9224	5413	7882	8990
Lipids	6448	5879	5729	5729

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[a] Data reported are means of one or more determinations made during the steady-state period.

[b] The feed slurry for this run was pretreated with cellulase (Novo Celluclast 1.5L) and cellobiase (Novozym 188) at dosages of 2.76 g/kg feed TS and 0.28 g/kg feed TS, respectively. Lipase (Novozym 225) was added to the acid digester at a dosage of 2.75 g/kg feed TS.

[c] System effluent qualities are the same as those of the methane digester.

[d] Numbers in parentheses are the coefficients of variation, expressed as the percent ratio of standard deviation to the mean.

TABLE 107. EFFECT OF CELLULASE-CELLOBIASE AND LIPASE TREATMENT ON STEADY-STATE ORGANIC REDUCTION EFFICIENCIES OF MESO-MESO CFCSTR TWO-PHASE SYSTEMS OPERATED WITH MIXED DOWNERS GROVE PRIMARY AND STICKNEY ACTIVATED SLUDGES AT ABOUT A 3-DAY HRT[a]

	Two-phase digestion with untreated raw sludge			Two-phase digestion with enzyme treated sludge[b]		
	Acid digester	Methane digester	System	Acid digester	Methane digester	System
Run number	---------- TP3M-M ----------			-------- TP3M-M(E) ---------		
HRT, days	0.91	2.15	3.06	0.93	2.31	3.24
VS reduction, %						
MOP_{16}[c]	17.1	11.4	26.5	14.2	13.0	25.4
Wt-of-gas-basis[d]	13.5	26.1	35.5	14.5	30.3	39.5
Carbon-in-gas basis[e]	10.1	18.4	28.5	11.4	21.6	33.0
Based on theoretical gas yield[f]	10.0	18.3	28.3	11.2	21.3	32.6
Biodegradable VS reduction[g]	17.3	31.5	48.8	19.4	36.8	56.1
Reduction of organic components, %						
Crude protein	33.0	12.7	41.5	--	--	--
Carbohydrates	15.8	41.3	50.6	49.9	27.3	63.6
Lipids	19.7	8.8	26.8	4.1	36.3	38.9

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[a] Data reported are means of one or more determinations made during the steady-state period.

[b] The feed slurry for this run was pretreated with cellulase (Novo Celluclast 1.5L) and cellobiase (Novozym 188) at dosages of 2.76 g/kg feed TS and 0.28 g/kg feed TS, respectively. Lipase (Novozym 225) was added to the acid digester at a dosage of 2.75 g/kg feed TS.

[c] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times (VS_i - VS_o)/[VS_i - (VS_i \times VS_o)]$.

[d] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times$ (wt of product gases)/(wt of VS fed).

[e] These VS reductions were calculated according to the following formula:
$VS_R = 100 \times$ (1.84 X mass flow rate of product gas carbon)/mass flow rate of VS fed.

[f] These VS reductions were calculated by expressing the observed total gas yield as a percentage of the theoretical gas yield of 1.078 SCM/kg VS added.

[g] The biodegradable VS reductions were calculated by dividing the theoretical-gas-yield-based VS reduction by a biodegradability factor of 0.58.

Overall, cellulase-cellobiase and lipase treatments of sludge increased the methane yield from the mesophilic CFCSTR two-phase system by about 23% over that obtained with untreated feeds. This increase is significant at the low HRT level of 3 days selected for the tests.

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APPENDIX A

FEED SLURRY ANALYSES

TABLE A-1. DIGESTER FEED SLURRY SOLIDS ANALYSES*

Digester feed prepared from sludge lot/batch nos.	Run in which used	Digester no(s).	SS/ SS/NSS†	Sample date(s)+	Total solids		Volatile solids		Fixed solids (by diff)	
					mg/L	wt %	mg/L	wt % of TS	mg/L	wt % of TS
1/8	SS15M	331	SS	1/6/83	45,100	4.51	33,500	74.28	11,600	25.72
1/14	SS15M	331	NSS	3/10/83	50,740	5.07	36,720	72.37	14,020	27.63
4/2	SS7M	331	NSS	7/25/83	64,390	6.44	47,010	73.01	17,380	26.99
4/2	SS7M	331	NSS	7/27/83	65,760	6.58	49,190	74.80	16,570	25.20
5/2	TP15M-M	332-333	NSS	8/24/83	45,810	4.58	33,410	72.93	12,400	27.07
5/2	TP15M-M	332-333	NSS	8/26/83	46,000	4.60	32,750	71.20	13,250	28.80
5/3	AP2M7	334	NSS	10/3/83	71,850	7.19	50,160	69.81	21,690	30.19
5/3	AP2T7	335	SS	10/3/83	77,930	7.79	53,440	68.57	24,490	31.43
5/3	AP2M7	334	NSS	10/14/83	67,420	6.74	47,430	70.35	19,990	29.65
5/3	AP2T7	335	SS	10/14/83	71,600	7.16	50,280	70.22	21,320	29.78
5/4	AP2M7	334	NSS	10/26/83	74,380	7.44	48,780	65.58	25,600	34.42
5/4	AP2T7	335	SS	10/26/83	77,710	7.77	52,620	67.71	25,090	32.29
5/4	TP15M-M	332-333	SS	11/5/83	40,280	4.03	28,660	71.15	11,620	28.85
5/4	AP2T7	335	SS	11/9/83	82,170	8.22	53,860	65.55	28,310	34.45
5/4	TP15M-M	332-333	SS	11/11/83-11/14/83	39,350	3.94	28,100	71.41	11,250	28.59
6/1	TP15M-M	332-333	NSS	11/23/83	44,260	4.43	30,140	68.10	14,120	31.90
6/1	TP15M-H	332-333	NSS	11/28/83	43,950	4.40	30,040	68.35	13,910	31.65
6/1	TP15M-M	332-333	NSS	11/25/83	43,610	4.36	30,240	69.34	13,370	30.66
6/1	TP15M-M	332-333	NSS	11/25/83-11/27/83	41,800	4.18	28,130	67.30	13,670	32.70
6/1	TP15M-M	332-333	NSS	11/26/83	42,630	4.26	30,240	70.94	12,390	29.06
6/1	TP15M-M	332-333	NSS	11/27/83	42,650	4.27	29,660	69.54	12,990	30.46
6/1	TP15M-M	332-333	NSS	11/28/83	39,690	3.97	27,880	70.24	11,810	29.76
6/1	TP15M-H	332-333	NSS	11/30/83	34,210	3.42	24,150	70.59	10,060	29.41
6/2	SS7M	331	SS	12/9/83	64,080	6.41	43,950	68.59	20,130	31.41
6/2	AP2M7	334	NSS	12/9/83	70,160	7.02	49,480	70.52	20,680	29.48
6/2	SS7M	331	SS	12/9/83-12/11/83	67,300	6.73	45,400	67.46	21,900	32.54
6/2	AP2M7	334	SS	12/9/83-2/11/83	68,580	6.86	47,160	68.77	21,420	31.23
6/2	AP2M7	334	SS	12/9/83-12/14/83	69,460	6.95	46,930	67.56	22,530	32.44
6/2	SS7M	331	SS	12/12/83-12/14/83	75,760	7.58	49,750	65.67	26,010	34.33
6/2	AP2M7	334	SS	12/12/83-12/14/83	70,430	7.04	46,700	66.31	23,730	33.69
6/2	SS7M	331	SS	12/31/83-1/9/84	80,340	8.03	53,580	66.69	26,760	33.31
6/4	SS7M	331	NSS	1/10/84-1/19/84	56,310	5.63	36,530	64.87	19,780	35.13
7/4	SS3M	331	NSS	3/3/84-3/12/84	65,740	6.57	50,650	77.05	15,090	22.95

(continued)

TABLE A-1 (continued)

Digester feed prepared from sludge lot/batch nos.	Run in which used	Digester no(s).	SS/ NSS[†]	Sample date(s)[+]	Total solids		Volatile solids		Fixed solids (by diff)	
					mg/L	wt %	mg/L	wt % of TS	mg/L	wt % of TS
8/1	SS3M	331	NSS	3/7/84	62,290	6.23	48,280	77.51	14,010	22.49
8/1	TP7M-M(NG)	332-333	NSS	3/7/84	61,670	6.17	47,920	77.70	13,750	22.30
8/1	AP1.3M7	334	NSS	3/7/84	59,710	5.97	46,290	77.52	13,420	22.48
8/1	AP1.3T7	335	NSS	3/7/84	59,770	5.98	46,400	77.63	13,370	22.37
8/5	AP1.3T7	335	SS	3/18/84-3/27/84	55,960	5.60	42,890	76.64	13,070	23.36
8/5	SS3M	331	SS	3/28/84-4/6/84	63,020	6.30	47,940	76.07	15,080	23.93
8/5	AP1.3M7	334	SS	3/28/84-4/6/84	59,380	5.94	44,960	75.72	14,420	24.28
12/1	SS15T	337	SS	5/31/84-6/9/84	41,560	4.16	31,630	76.11	9,930	23.89
13/1	AP2T5	335	SS	6/12/84-6/16/84	65,310	6.53	49,840	76.31	15,470	23.69
13/1	AP2M5	334	SS	6/19/84-6/23/84	67,530	6.75	51,460	76.20	16,070	23.80
13/1	UTP7M-M	338-339	SS	6/28/84-7/1/84	69,870	6.99	52,230	74.75	17,640	25.25
13/1	AP1.3T5	335	SS	6/28/84-7/2/84	67,530	6.75	50,750	75.15	16,780	24.85
14/1	TP7M-M(NG)	332-333	NSS	7/1/84	67,230	6.72	48,220	71.72	19,010	28.28
14/1	AP1.3M5	334	SS	7/2/84-7/6/84	68,440	6.84	47,970	70.09	20,470	29.91
16/1	SS7T	331	SS	8/2/84-8/6/84	67,250	6.73	49,740	73.96	17,510	26.04
16/1	SS3T	335	SS	8/8/84-8/12/84	67,510	6.75	49,240	72.94	18,270	27.06
16/1	TP15M-T	334-337	SS	8/8/84-8/12/84	47,060	4.71	32,900	69.91	14,160	30.09
16/1	SS3T	335	SS	8/8/84-8/17/84	66,735	6.67	48,615	72.85	18,120	27.15
16/1	TP15M-T	334-337	SS	8/8/84-8/17/84	46,045	4.60	32,420	70.41	13,625	29.59
16/1	SS3T	335	SS	8/13/84-8/17/84	65,960	6.60	47,990	72.76	17,970	27.24
16/1	TP15M-T	334-337	SS	8/13/84-8/17/84	45,030	4.50	31,940	70.93	13,090	29.07
17/1	TP7M-T	334-331	SS	9/10/84-9/14/84	66,430	6.64	48,640	73.22	17,790	26.78
17/1	TP7M-M(NG)	332-333	NSS	9/1/84-9/15/84	68,960	6.90	50,640	73.43	18,320	26.57
17/1	TP7M-M	334-333	SS	9/25/84-9/29/84	66,540	6.65	48,860	73.43	17,680	26.57
28/1	TP3M-M	334-333	SS	12/17/84-12/21/84	68,500	6.85	46,600	68.03	21,900	31.97
32/1	TP3M-M(E)	334-333	SS	1/8/85-1/12/85	69,470	6.95	47,420	68.26	22,050	31.74

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[*] Data reported are the averages of triplicate determinations.

[+] 'SS' means the sample was collected during steady-state operation; 'NSS' means the sample was collected during non-steady-state operation.

[†] A single sample data indicates that the analysis was conducted on a grab sample collected that day. A time period under this column indicates the start and end dates of collection of a grab or time-composite sample used for analysis.

TABLE A-2. DIGESTER FEED SLURRY SUSPENDED SOLIDS ANALYSES*

Digester feed prepared from sludge lot/batch nos.	Run in which used	Digester no(s).	SS/ NNS‡	Sample date(s)†	Total solids		Volatile solids		Fixed solids (by diff)		Total suspended solids		Volatile suspended solids		Fixed suspended solids (by diff)	
					mg/L	wt %	mg/L	wt % of TS	mg/L	wt % of TS	mg/L	wt % of TS	mg/L	wt % of TS	mg/L	wt % of TS
1/14	SS15M	331	NSS	3/10/83	50,740	5.07	36,720	72.37	14,020	27.63	48,000	94.60	36,270	71.48	11,730	23.12
5/3	AP2T7	335	SS	10/14/83	71,600	7.16	50,280	70.22	21,320	29.78	65,740	91.82	44,710	62.44	21,030	29.37
5/4	AP2M7	334	NSS	10/26/83	74,380	7.44	48,780	65.58	25,600	34.42	65,160	87.60	41,800	56.20	23,360	31.41
5/4	AP2T7	335	SS	10/26/83	77,710	7.77	52,620	67.71	25,090	32.29	69,890	89.94	45,000	57.91	24,890	32.03
5/4	TP15M-M	332-333	SS	11/5/83	40,280	4.03	28,660	71.15	11,620	28.85	33,560	83.32	27,100	67.28	6,460	16.04
5/4	TP15M-M	332-333	SS	11/11/83-11/14/83	39,350	3.94	28,100	71.41	11,250	28.59	32,100	81.58	25,910	65.84	6,190	15.73
6/2	AP2M7	334	SS	12/9/83-12/14/83	69,460	6.95	46,930	67.56	22,530	32.44	62,170	89.50	45,690	65.78	16,480	23.73
6/2	SS7M	331	SS	12/31/83-1/9/84	80,340	8.03	53,580	66.69	26,760	33.31	63,650	79.23	47,670	59.34	15,980	19.89
8/5	AP1.3T7	335	SS	3/18/84-3/27/84	55,960	5.60	42,890	76.64	13,070	23.36	43,740	78.16	36,700	65.58	7,040	12.58
8/5	AP1.3M7	334	SS	3/28/84-4/6/84	59,380	5.94	44,960	75.72	14,420	24.28	46,140	77.70	39,200	66.02	6,940	11.69

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* Data reported are the averages of triplicate determinations.

† 'SS' means the sample was collected during steady-state operation; 'NSS' means the sample was collected during non-steady-state operation.

‡ A single sample date indicates that the analysis was conducted on a grab sample collected that day. A time period under this column indicates the start and end dates of collection of a grab or time-composite sample used for analysis.

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TABLE A-3. DIGESTER FEED SLURRY pH, AMMONIA NITROGEN, AND ALKALINITY ANALYSES*

Digester feed prepared from sludge lot/batch nos.	Run in which used	Digester no(s).	SS/ NSS†	Sample date(s)‡	pH	Ammonia nitrogen, mg/L	Total alkalinity, mg/L as $CaCO_3$	Bicarbonate alkalinity, mg/L as $CaCO_3$
1/8	SS15M	331	SS	1/6/83	—	280	—	—
1/9	SS15M	331	SS	1/18/83	6.84	—	4583	3912
1/9	SS15M	331	SS	1/20/83	6.64	—	4700	4029
4/2	NS7M	331	NSS	7/25/83	—	569	—	—
4/2	TP15M-M	332-333	NSS	7/25/83	—	385	—	—
5/1	AP2M7	334	NSS	9/22/83	—	247	—	—
5/3	AP2M7	334	NSS	10/3/83	6.48	—	5200	4318
5/3	AP2T7	335	SS	10/3/83	6.20	299	5050	4106
5/3	AP2M7	334	NSS	10/14/83	—	224	—	—
5/3	AP2T7	335	SS	10/14/83	—	268	—	—
5/4	AP2T7	335	SS	11/9/83	6.43	168	5600	4855
5/4	TP15M-M	332-333	SS	11/11/83-11/14/83	—	131	—	—
6/1	TP15M-M	332-333	SS	11/28/83	7.19	—	3140	2838
6/1	AP2T7	335	NSS	11/30/83	6.58	—	4720	4352
6/1	TP15M-M	332-333	SS	12/2/83	7.00	—	3185	2883
6/2	AP2M7	334	SS	12/9/83-12/14/83	—	219	—	—
6/2	SS7M	331	SS	12/14/83	6.42	—	5390	4334
6/2	AP2M7	334	SS	12/14/83	6.61	—	5373	4510
6/2	SS7M	331	SS	12/31/83-1/9/84	—	354	—	—
8/5	AP1.3T7	335	SS	3/18/84-3/27/84	—	509	—	—
8/5	AP1.3T7	335	SS	3/20/84	6.01	—	5200	3091
8/5	SS3M	331	SS	3/28/84-4/6/84	—	589	—	—
8/5	AP1.3M7	334	SS	3/28/84-4/6/84	—	624	—	—
8/5	SS3M	331	SS	4/1/84	6.05	—	4575	2455
8/5	AP1.3M7	334	SS	4/1/84	6.16	—	4375	1933
12/1	SS15T	337	SS	5/31/84-6/9/84	—	237	—	—
12/1	SS15T	337	SS	6/10/84	5.94	—	3250	2160
13/1	AP2T5	335	SS	6/12/84-6/16/84	—	337	—	—
13/1	AP2M5	334	SS	6/17/84	5.86	—	4820	2844
13/1	AP2T5	335	SS	6/17/84	5.85	—	4870	3125
13/1	AP2M5	334	SS	6/19/84-6/23/84	—	302	—	—
13/1	UTP7M-M	338-339	SS	6/28/84-7/1/84	—	327	—	—
13/1	AP1.3T5	335	SS	6/28/84-7/2/84	—	321	—	—
13/1	UTP7M-M	338-339	SS	7/6/84	6.04	—	5500	3766
13/1	AP1.3T5	335	SS	7/6/84	5.87	—	7340	5665

(continued)

TABLE A-3 (continued)

Digester feed prepared from sludge lot/batch nos.	Run in which used	Digester no(s).	SS/ NSS†	Sample date(s)+	pH	Ammonia nitrogen, mg/L	Total alkalinity, mg/L as $CaCO_3$	Bicarbonate alkalinity, mg/L as $CaCO_3$
14/1	AP1.3M5	334	SS	7/2/84-7/6/84	--	369	--	--
14/1	AP1.3M5	334	SS	7/6/84	6.16	--	7175	5513
16/1	SS7T	331	SS	7/30/84	6.16	--	6850	3845
16/1	SS7T	331	SS	8/2/84-8/6/84	--	587	--	--
16/1	SS3T	335	SS	8/8/84-8/17/84	--	755	--	--
16/1	TP15M-T	334-337	SS	8/8/84-8/17/84	--	528	--	--
16/1	SS3T	335	SS	8/16/84	6.37	--	6105	2737
16/1	TP15M-T	334-337	SS	8/17/84	6.52	--	4618	2343
17/1	TP7M-T	334-331	SS	9/10/84-9/14/84	--	546	--	--
17/1	TP7M-M(NG)	332-333	NSS	9/11/84-9/15/84	--	585	--	--
17/1	TP7M-M(NG)	332-333	NSS	9/13/84	6.00	--	5250	3473
17/1	TP7M-T	334-331	SS	9/13/84	6.33	--	6050	4421
17/1	TP7M-M	334-333	SS	9/25/84-9/29/84	--	582	--	--
17/1	TP7M-M	334-333	SS	9/29/84	6.37	--	5850	3863
28/1	TP3M-M	334-333	SS	12/17/84-12/21/84	--	535	--	--
28/1	TP3M-M	334-333	SS	12/19/84	6.06	--	4750	1245

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* Data reported are the averages of duplicate or triplicate determinations.

† 'SS' means the sample was collected during steady-state operation; 'NSS' means the sample was collected during non-steady-state operation.

+ A single sample data indicates tha the analysis was conducted on a grab sample collected that day. A time period under this column indicates the start and end dates of collection of a grab or time-composite sample used for analysis.

TABLE A-4. DIGESTER FEED SLURRY TOTAL AND FILTRATE CHEMICAL OXYGEN DEMAND (COD) ANALYSES*

Digester feed prepared from sludge lot/batch nos.	Run in which used	Digester no(s).	SS/ NSS†	Sample date(s)+	Total COD, mg/L	Total COD, g/g VS	Filtrate COD, mg/L	Filtrate COD, g/g VS	Particulate (by diff) COD, mg/L	Particulate (by diff) COD, g/g VS
1/14	SS15M	331	NSS	3/10/83	48,270	1.315	5324	0.145	42,946	1.170
5/1	AP2M7	334	NSS	9/22/83	--	--	6390	0.131	--	--
5/3	AP2M7	334	NSS	10/3/83	--	--	4922	0.098	--	--
5/3	AP2M7	334	NSS	10/3/83	79,530	1.586	4696	0.094	74,834	1.492
5/3	AP2T7	335	SS	10/3/83	--	--	4979	0.093	--	--
5/3	AP2T7	335	SS	10/3/83	87,096	1.630	4918	0.092	82,178	1.538
5/3	AP2M7	334	NSS	10/14/83	66,723	1.407	5673	0.120	61,050	1.287
5/3	AP2T7	335	SS	10/14/83	78,047	1.552	6547	0.130	71,500	1.422
5/4	AP2M7	334	NSS	10/26/83	78,798	1.615	--	--	--	--
5/4	TP15M-M	332-333	SS	11/5/83	39,580	1.381	2745	0.096	36,835	1.285
5/4	TP15M-M	332-333	SS	11/11/83-11/14/83	34,822	1.239	2601	0.093	32,221	1.147
6/2	AP2M7	334	SS	12/9/83-12/14/83	77,702	1.656	4605	0.098	73,097	1.558
6/2	SS7M	331	SS	12/31/83-1/9/84	80,617	1.505	4211	0.079	76,406	1.426
8/5	AP1.3T7	335	SS	3/18/84-3/27/84	76,394	1.781	7042	0.164	69,352	1.617
8/5	SS3M	331	SS	3/28/84-4/6/84	85,983	1.794	7968	0.166	78,015	1.627
8/5	AP1.3M7	334	SS	3/28/84-4/6/84	81,896	1.822	7753	0.172	74,143	1.649

* Data reported are the averages of two or more determinations.

† 'SS' means the sample was collected during steady-state operation; 'NSS' means the sample was collected during non-steady-state operation.

+ A single sample date indicates that the analysis was conducted on a grab sample collected that day. A time period under this column indicates the start and end dates of collection of a grab or time-composite sample used for analysis.

TABLE A-5. DIGESTER FEED SLURRY AMMONIA, ORGANIC, AND TOTAL KJELDAHL NITROGEN ANALYSES*

Digester feed prepared from sludge lot/batch nos.	Run in which used	Digester no(s).	SS/NSS†	Sample date(s)+	Ammonia nitrogen			Organic nitrogen			Total Kjeldahl nitrogen		
					mg/L	wt % of TS	wt % of VS	mg/L	wt % of TS	wt % of VS	mg/L	wt % of TS	wt % of VS
1/8	SS15M	331	SS	1/6/83	280	0.62	0.84	1971	4.37	5.88	2251	4.99	6.72
4/2	SS7M	331	NSS	7/25/83	569	0.88	1.21	--	--	--	--	--	--
4/2	TP15M-M	332-333	NSS	7/25/83	385	0.84	1.15	--	--	--	--	--	--
5/1	AP2M7	334	NSS	9/22/83	247	0.35	0.51	2352	3.38	4.82	2599	3.73	5.33
5/3	AP2T7	335	SS	10/3/83	299	0.38	0.56	2245	2.88	4.20	2544	3.26	4.76
5/3	AP2M7	334	NSS	10/14/83	224	0.33	0.47	2156	3.20	4.55	2380	3.53	5.02
5/3	AP2T7	335	SS	10/14/83	268	0.37	0.53	2098	2.93	4.17	2366	3.30	4.71
5/4	AP2T7	335	SS	11/9/83	168	0.20	0.31	2520	3.07	4.68	2688	3.27	4.99
5/4	TP15M-M	332-333	SS	11/11/83-11/14/83	131	0.33	0.47	1456	3.70	5.18	1587	4.03	5.65
6/2	AP2M7	334	SS	12/9/83-12/14/83	219	0.32	0.47	2044	2.94	4.36	2263	3.26	4.82
6/2	SS7M	331	SS	12/31/83-1/9/84	354	0.44	0.66	2347	2.92	4.38	2701	3.36	5.04
8/5	AP1.3T7	335	SS	3/18/84-3/27/84	509	0.91	1.19	2420	4.32	5.64	2929	5.23	6.83
8/5	SS3M	331	SS	3/28/84-4/6/84	589	0.93	1.23	2617	4.15	5.46	3206	5.09	6.69
8/5	AP1.3M7	334	SS	3/28/84-4/6/84	624	1.05	1.39	2563	4.32	5.70	3187	5.37	7.09
12/1	SS15T	337	SS	5/31/84-6/9/84	237	0.57	0.75	1867	4.49	5.90	2104	5.06	6.65
13/1	AP2T5	335	SS	6/12/84-6/16/84	337	0.52	0.68	2918	4.47	5.85	3255	4.98	6.53
13/1	AP2M5	334	SS	6/19/84-6/23/84	302	0.45	0.59	2820	4.18	5.48	3122	4.62	6.07
13/1	UTP7M-M	338-339	SS	6/28/84-7/1/84	327	0.47	0.63	2793	4.00	5.35	3120	4.47	5.97
13/1	AP1.3T5	335	SS	6/28/84-7/2/84	321	0.48	0.63	2933	4.34	5.78	3254	4.82	6.41
14/1	AP1.3M5	334	SS	7/2/84-7/6/84	369	0.54	0.77	2335	3.41	4.87	2704	3.95	5.64

(continued)

TABLE A-5 (continued)

Digester feed prepared from sludge lot/batch nos.	Run in which used	Digester no(s).	SS/ NSS[†]	Sample date(s)[+]	Ammonia nitrogen			Organic nitrogen			Total Kjeldahl nitrogen		
					mg/L	wt % of TS	wt % of VS	mg/L	wt % of TS	wt % of VS	mg/L	wt % of TS	wt % of VS
16/1	SS7T	331	SS	8/2/84-8/6/84	587	0.87	1.18	2750	4.09	5.53	3337	4.96	6.71
16/1	SS3T	335	SS	8/8/84-8/17/84	755	1.13	1.55	2779	4.16	5.72	3534	5.30	7.27
16/1	TP15M-T	334-337	SS	8/8/84-8/17/84	528	1.15	1.63	1912	4.15	5.90	2440	5.30	7.53
17/1	TP7M-T	334-331	SS	9/10/84-9/14/84	546	0.82	1.12	2662	4.01	5.47	3208	4.83	6.60
17/1	TP7M-M(NC)	332-333	NSS	9/11/84-9/15/84	585	0.85	1.16	2482	3.60	4.90	3067	4.45	6.06
17/1	TP7M-M	334-333	SS	9/25/84-9/29/84	582	0.87	1.19	2450	3.68	5.01	3032	4.56	6.21
28/1	TP3M-M	334-333	SS	12/17/84-12/21/84	535	0.78	1.15	2876	4.20	6.17	3411	4.98	7.32

[*] Data reported are the averages of duplicate or triplicate determinations.

[†] 'SS' means the sample was collected during steady-state operation; 'NSS' means the sample was collected during non-steady-state operation.

[+] A single sample date indicates that the analysis was conducted on a grab sample collected that day. A time period under this column indicates the start and end dates of collection of a grab or time-composite sample used for anaysis.

TABLE A-6. ORGANIC COMPONENT ANALYSES FOR DIGESTER FEED SLURRIES[a]

Feed designation	Run in which used*	SS/NSS[b]	Digester no(s).[c]	Sample date(s)	TS	VS		Crude protein		Total carbohydrate		Lipids	
Lot no./batch no.					mg/L	mg/L	wt % of TS	mg/L	wt % of VS	mg/L	wt % of VS	mg/L	wt % of VS
1/8	SS15M	SS	331	1/6/83	45,100	33,500	74.27	12,315	36.76	6532	19.49	9288	27.72
	SS15M	SS	331	1/6/83	45,100	33,500	74.27	--	--	--	--	6700	20.00
	SS15M	SS	331	1/6/83	45,100	33,500	74.27	--	--	--	--	7239	21.60
							Means		**36.76**		**19.49**		**23.11**
3/2 (diluted)[d]	--	NSS	332-333	6/4/83	53,500	37,434	69.97	--	--	--	--	9160	24.47
4/1 (diluted)	--	NSS	332-333	7/11/83	71,700	51,803	72.25	--	--	--	--	16,294	31.45
4/2 (diluted)	--	NSS	331	7/25/83	64,390	47,010	73.00	--	--	--	--	14,100	29.99
	--	NSS	331	7/27/83	65,760	49,190	74.80	--	--	--	--	17,933	36.45
	--	NSS	331	7/27/83	65,760	49,190	74.80	--	--	--	--	15,300	31.10
	--												
							Means						**32.51**
5/1 (diluted)	--	NSS	334	9/22/83	71,850	50,160	69.81	14,700	29.31	--	--	--	--
	--	NSS	334	9/22/83	67,420	47,430	70.35	14,700	37.99	--	--	--	
5/3 (diluted)	--	NSS	334	10/3/83	71,850	50,160	69.81	--	--	13,567	27.04	13,400	26.71
	AP2T7	SS	335	10/3/83	77,930	53,440	68.57	14,031	26.25	14,100	26.38	14,600	27.32
	--	NSS	334	10/14/83	67,420	47,430	70.35	13,475	28.41	12,350	26.03	--	--
	AP2T7	SS	335	10/14/83	71,600	50,280	70.22	13,112	26.07	13,300	26.45	14,100	28.04
5/4 (diluted)	AP2T7	SS	335	11/9/83	82,170	53,860	65.54	15,750	29.24	--	--	--	--
	TP15M-M	SS	332-333	11/10/83	39,350	28,100	71.41	--	--	--	--	6,200	22.06
	TP15M-M	SS	332-333	11/11-11/14/83	39,350	28,100	71.41	9100	32.38	8307	29.56	--	--
	TP15M-M	SS	332-333	11/13/83	39,350	28,100	71.41	--	--	--	--	7,450	26.51
							Means		**30.81**		**29.56**		**24.28**
6/2 (diluted)	SS7M	SS	331	12/12-12/14/83	75,760	49,750	65.66	--	--	9,958	20.01	--	--
	SS7M	SS	331	12/31/83-1/9/84	80,340	53,580	66.69	14,669	27.37	9,036	16.86	18,929	35.32
	AP2M7	SS	334	12/9-12/14/83	69,460	46,930	67.56	12,775	27.22	--	--	19,068	40.63
							Means		**27.30**		**18.44**		**37.98**
8/5 (concentrated)	A1.3T7	SS	335	3/18-3/27/84	55,960	42,890	76.64	15,125	35.26	12,118	28.25	--	--
	SS3M	SS	331	3/28-4/6/84	63,020	47,940	76.07	16,536	34.11	12,012	25.05	12,392	25.85
	SS3M	SS	331	3/28-4/6/84	63,020	47,940	76.07	--	--	11,538	23.69	--	--
	AP1.3M7	SS	334	3/28-4/6/84	59,380	44,960	75.71	16,019	35.62	12,724	28.30	11,736	26.10
							Means		**35.00**		**26.32**		**25.98**
12/1 (diluted)	SS15T	SS	337	5/31-6/9/84	41,560	31,630	76.10	11,669	36.89	7,292	23.05	10,030	31.71
13/1 (diluted)	AP2T5	SS	335	6/12-6/16/84	65,310	49,840	76.31	18,238	36.59	--	--	--	--
	AP2M5	SS	334	6/19-6/23/84	67,530	51,460	76.20	17,625	34.24	10,994	21.36	15,104	29.35
	AP2M5	SS	334	6/19-6/23/84	67,530	51,460	76.20	--	--	11,813	22.95	--	--
	UTP7M-M	SS	338-339	6/28-7/1/84	69,870	52,230	74.75	--	--	11,956	22.89	--	--
	UTP7M-M	SS	338-339	6/28-7/1/84	69,870	52,230	74.75	17,456	33.42	13,934	26.67	--	--
	AP1.3T5	SS	335	6/28-7/2/84	67,530	50,750	75.15	18,331	36.12	10,786	21.25	--	--
	AP1.3T5	SS	335	6/28-7/2/84	67,530	50,750	75.15	--	--	12,016	23.67	--	--
							Means		**35.09**		**23.13**		**29.35**
14/1 (diluted)	AP1.3M5	SS	334	7/2-7/6/84	68,440	47,970	70.09	14,594	30.42	13,637	28.42	9,824	20.48

(Continued)

TABLE A-6 (continued)

Feed designation	Run in which used[a]	SS/NSS	Digester no.	Sample date(s)	TS	VS		Crude protein		Total carbohydrate		Lipids	
Lot No./Batch No.					mg/L	mg/L	wt % of TS	mg/L	wt % of VS	mg/L	wt % of VS	mg/L	wt % of VS
16/1 (diluted)	SS7T	SS	331	8/2-8/6/84	67,250	49,740	73.96	17,188	34.55	9,292	18.68	9,705	19.51
	SS7T	SS	331	8/2-8/6/84	67,250	49,740	73.96	--	--	10,238	20.58	--	--
	SS3T	SS	335	8/8-8/17/84	66,735	48,615	72.84	17,369	35.72	10,363	21.31	8,952	18.41
	SS3T	SS	335	8/8-8/17/84	66,735	48,615	72.84	--	--	8,497	17.47	--	--
	TP15M-T	SS	334-337	8/8-8/17/84	46,045	32,420	70.40	11,950	36.85	5,943	18.33	7,156	22.07
							Means		**35.71**		**19.27**		**20.00**
17/1 (diluted)	TP7M-T	SS	334-331	9/10-9/14/84	66,430	48,640	73.21	16,638	34.20	12,202	25.08	11,994	24.66
	TP7M-T	SS	334-331	9/10-9/14/84	66,430	48,640	73.21	--	--	11,114	22.84	--	--
	—	NSS	332-333	9/11-9/15/84	68,960	50,640	73.43	15,512	30.63	10,594	20.92	--	--
	TP7M-M	SS	334-333	9/25-9/29/84	66,540	48,860	73.42	15,312	31.33	11,718	22.87	9,314	19.06
	TP7M-M	SS	334-333	9/25-9/29/84	66,540	48,860	73.42	--	--	10,610	21.71	--	--
							Means		**32.05**		**22.68**		**21.86**
28/1 (diluted)	TP3M-M	SS	334-333	12/17-12/21/84	68,500	46,600	68.02	17,975	38.57	10,459	22.44	8,026	17.22
	TP3M-M	SS	334-333	12/17-12/21/84	68,500	46,600	68.02	--	--	11,452	24.57	--	--
							Means		**38.57**		**23.50**		**17.22**
32/1 (diluted)	TP3M-M(E)	SS	334-333	1/8-1/12/85	69,470	47,420	68.25	--	--	15,734	33.18	9,370	19.75

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[a] Data reported in this table were averages of replicate determinations.

[b] SS means that the run was at steady state during sampling; NSS is not steady state.

[c] One digester number indicates a single-stage digester. Two digester numbers indicate a two-phase system consisting of two digesters operated in series.

[d] Digester feed slurry was prepared by appropriately diluting the indicated feedstock.

APPENDIX B

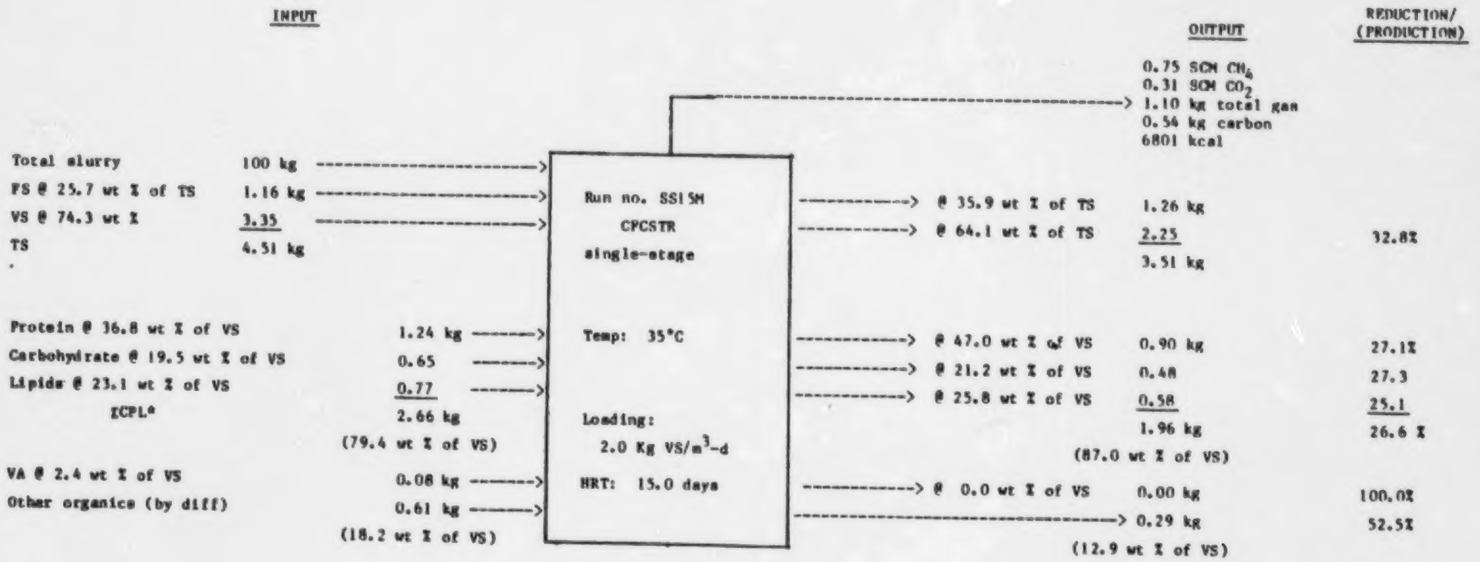
EFFLUENT ANALYSES FOR SINGLE-STAGE CFCSTR DIGESTERS

TABLE B-1. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. SS15M: CFCSTR SINGLE-STAGE MESOPHILIC (35°C) DIGESTER OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 15-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
1/2/83	--	30.59	0.71	68.69
1/8/83	--	29.66	0.27	70.07
1/10/83	--	29.63	0.65	69.72
1/14/83	--	28.73	1.04	70.23
1/29/83	--	29.00	0.00	71.00
2/5/83	--	29.63	0.63	69.75
2/8/83	--	28.93	0.48	70.59
2/10/83	0.00	29.15	0.00	70.85
2/18/83	0.00	28.89	0.81	70.30

TABLE B-2. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. SS15M CFCSTR SINGLE-STAGE MESOPHILIC (35°C) DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 15-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
12/31/82	0	0	0	0	0	0	0	0	--
1/8/83	0	0	0	0	0	0	0	0	--
1/14/83	0	0	0	0	0	0	0	0	--
1/21/83	0	0	0	0	0	0	0	0	--
1/28/83	0	0	0	0	0	0	0	0	--
2/5/83	2	0	0	0	0	0	0	2	7
2/8/83	0	0	0	0	0	0	0	0	0
2/12/83	3	0	0	0	0	0	0	3	3
2/19/83	0	0	0	0	0	0	0	0	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.75 SCM CH_4
0.31 SCM CO_2
1.10 kg total gas
0.54 kg carbon
6801 kcal
Total slurry 100 kg
FS @ 25.7 wt % of TS 1.16 kg
VS @ 74.3 wt % 3.35
TS 4.51 kg
Run no. SS15M
CFCSTR
single-stage
@ 35.9 wt % of TS 1.26 kg
@ 64.1 wt % of TS 2.25 32.8%
3.51 kg
Protein @ 36.8 wt % of VS 1.24 kg
Carbohydrate @ 19.5 wt % of VS 0.65
Lipids @ 23.1 wt % of VS 0.77
ΣCPL* 2.66 kg
(79.4 wt % of VS)
Temp: 35°C
@ 47.0 wt % of VS 0.90 kg 27.1%
@ 21.2 wt % of VS 0.48 27.3
@ 25.8 wt % of VS 0.58 25.1
1.96 kg 26.6 %
(87.0 wt % of VS)
Loading:
2.0 Kg VS/m³-d
HRT: 15.0 days
VA @ 2.4 wt % of VS 0.08 kg
Other organics (by diff) 0.61 kg
(18.2 wt % of VS)
@ 0.0 wt % of VS 0.00 kg 100.0%
0.29 kg 52.5%
(12.9 wt % of VS)

FS balance: effluent/feed = 109%

VS balance: effluent/feed = 100%

Carbon recovery in gas = 28%

Energy recovery in gas = 32%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

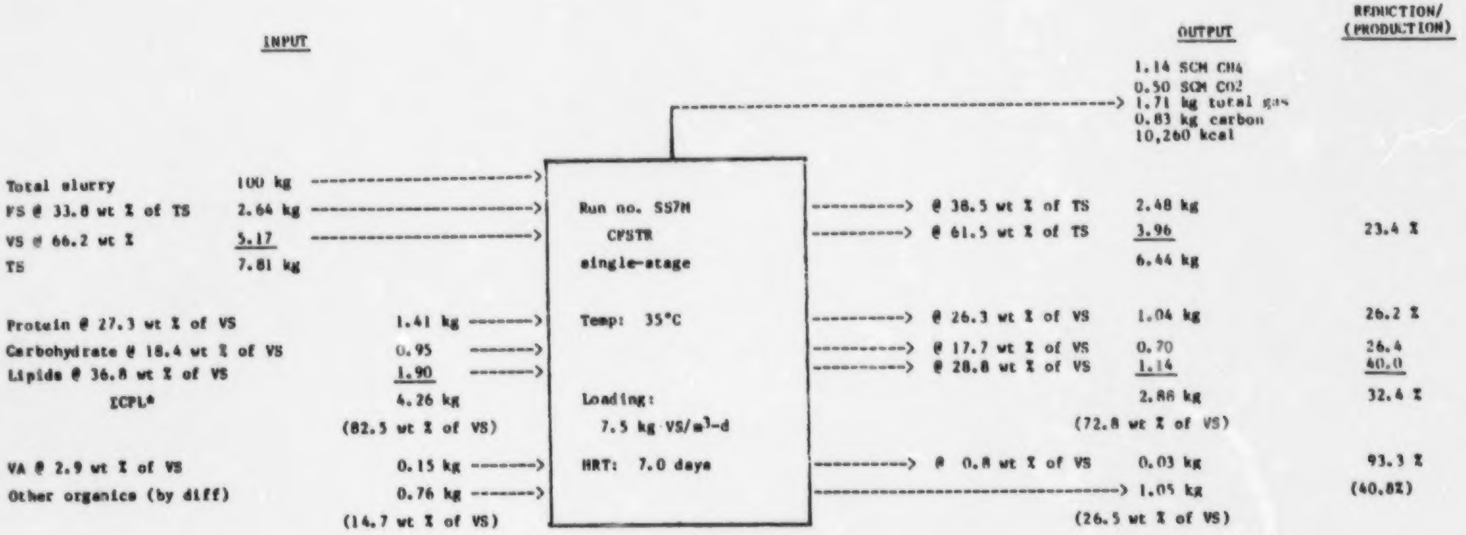
Figure B-1. Mass balances for single-stage CFCSTR mesophilic Run SS15M conducted with Hanover Park sludge at a 15-day HRT

TABLE B-3. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. SS7M: CFCSTR SINGLE-STAGE MESOPHILIC (35°C) DIGESTON OF HANOVER PARK SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
11/27/83	--	31.87	0.81	67.32
12/4/83	--	30.45	0.80	68.75
12/11/83	--	29.85	0.14	70.01
12/19/83	--	30.45	0.00	69.55
12/28/83	--	31.17	0.00	68.83
1/2/84	--	30.19	0.00	69.81

TABLE B-4. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. SS7M CFCSTR SINGLE-STAGE MESOPHILIC (35°C) DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
11/28/83	189	117	0	0	0	0	0	284	0
12/5/83	170	128	0	0	0	0	0	274	0
12/12/83	136	88	0	0	0	0	0	207	0
12/19/83	121	99	0	0	0	0	0	201	0
12/26/83	95	86	0	0	0	0	0	164	17
1/3/84	272	105	2	0	0	0	0	359	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
Total slurry 100 kg
FS @ 33.8 wt % of TS 2.64 kg
VS @ 66.2 wt % 5.17
TS 7.81 kg
Protein @ 27.3 wt % of VS 1.41 kg
Carbohydrate @ 18.4 wt % of VS 0.95
Lipids @ 36.8 wt % of VS 1.90
ΣCPL* 4.26 kg
(82.5 wt % of VS)
VA @ 2.9 wt % of VS 0.15 kg
Other organics (by diff) 0.76 kg
(14.7 wt % of VS)
Run no. SS7M
CFSTR
single-stage
Temp: 35°C
Loading:
7.5 kg VS/m³-d
HRT: 7.0 days
1.14 SCM CH4
0.50 SCM CO2
1.71 kg total gas
0.83 kg carbon
10,260 kcal
@ 38.5 wt % of TS 2.48 kg
@ 61.5 wt % of TS 3.96 23.4 %
6.44 kg
@ 26.3 wt % of VS 1.04 kg 26.2 %
@ 17.7 wt % of VS 0.70 26.4
@ 28.8 wt % of VS 1.14 40.0
2.88 kg 32.4 %
(72.8 wt % of VS)
@ 0.8 wt % of VS 0.03 kg 93.3 %
1.05 kg (40.8%)
(26.5 wt % of VS)

FS balance: effluent/feed = 94%
VS balance: effluent/feed = 110%
Carbon recovery in gas = 37%
Energy recovery in gas = 31%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

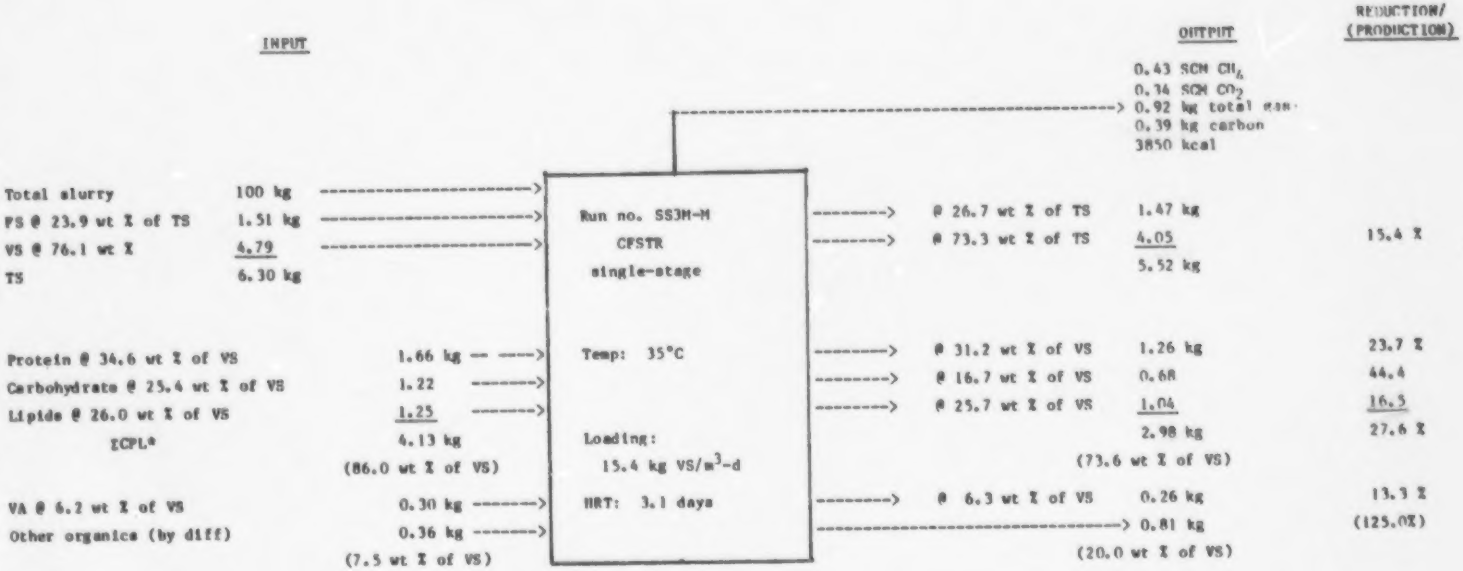
Figure B-2. Mass balances for single-stage CFCSTR mesophilic Run SS7M conducted with Hanover Park sludge at a 7-day HRT

TABLE B-5. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. SS3M: CFCSTR SINGLE-STAGE MESOPHILIC (35°C) DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 3-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
3/25/84	--	45.19	0.50	54.31
3/30/84	--	44.43	0.50	55.07
4/3/84	--	40.58	0.50	58.92
4/8/84	--	42.56	0.47	56.98

TABLE B-6. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. SS3M CFCSTR SINGLE-STAGE MESOPHILIC (35°C) DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 3-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
3/23/84	610	1,520	267	91	486	114	0	2,439	0
3/30/84	350	1,600	205	48	360	96	0	2,087	0
3/31/84	389	1,727	220	52	334	97	0	2,227	0
4/4/84	247	1,766	176	0	232	61	0	1,971	0
4/9/84	121	1,243	85	2	234	62	0	1,363	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.43 SCM CH4
0.34 SCM CO2
0.92 kg total gas
0.39 kg carbon
3850 kcal
Total slurry 100 kg
FS @ 23.9 wt % of TS 1.51 kg
VS @ 76.1 wt % 4.79
TS 6.30 kg
Run no. SS3M-M
CFSTR
single-stage
@ 26.7 wt % of TS 1.47 kg
@ 73.3 wt % of TS 4.05 15.4 %
5.52 kg
Protein @ 34.6 wt % of VS 1.66 kg
Carbohydrate @ 25.4 wt % of VS 1.22
Lipids @ 26.0 wt % of VS 1.25
ΣCPL* 4.13 kg
(86.0 wt % of VS)
Temp: 35°C
Loading:
15.4 kg VS/m3-d
HRT: 3.1 days
@ 31.2 wt % of VS 1.26 kg 23.7 %
@ 16.7 wt % of VS 0.68 44.4
@ 25.7 wt % of VS 1.04 16.5
2.98 kg 27.6 %
(73.6 wt % of VS)
VA @ 6.2 wt % of VS 0.30 kg
Other organics (by diff) 0.36 kg
(7.5 wt % of VS)
@ 6.3 wt % of VS 0.26 kg 13.3 %
0.81 kg (125.0%)
(20.0 wt % of VS)

FS balance: effluent/feed = 97%
VS balance: effluent/feed = 104%
Carbon recovery in gas = 14%
Energy recovery in gas = 12%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

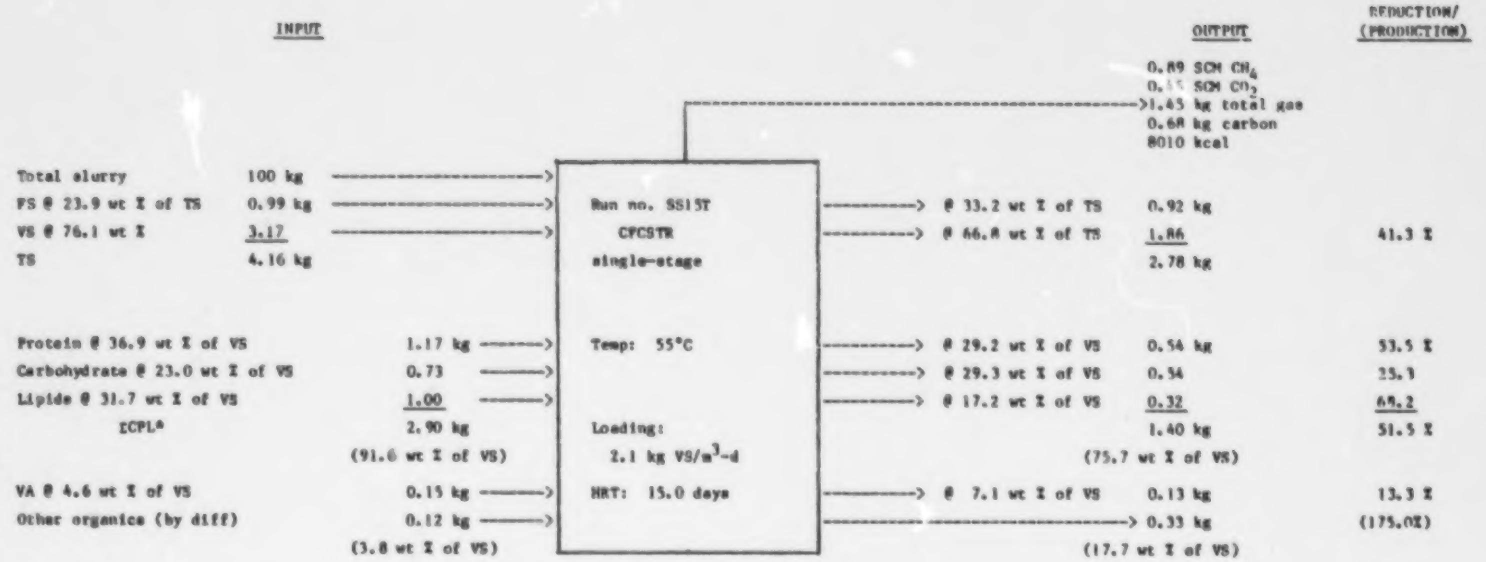
Figure B-3. Mass balances for single-stage CFCSTR mesophilic Run SS3M conducted with Hanover Park sludge at a 3-day HRT

TABLE B-7. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. SS15T: CFCSTR SINGLE-STAGE MESOPHILIC (55°C) DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 15-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
5/27/84	—	34.36	0.11	65.53
6/3/84	—	33.39	0.28	66.34
6/1/84	—	33.38	0.13	66.49
6/11/84	—	32.91	0.21	66.88

TABLE B-8. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. SS15T CFCSTR SINGLE-STAGE MESOPHILIC (55°C) DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 15-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
5/27/84	209	1,043	121	6	258	0	11	1,299	0
6/3/84	162	864	89	5	427	0	25	1,191	0
6/9/84	118	746	33	0	37	16	0	776	0
6/11/84	128	721	34	0	234	16	0	882	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.89 SCM CH_4
0.45 SCM CO_2
1.45 kg total gas
0.68 kg carbon
8010 kcal
Total slurry
100 kg
FS @ 23.9 wt % of TS
0.99 kg
VS @ 76.1 wt %
3.17
TS
4.16 kg
Run no. SS15T
CFCSTR
single-stage
@ 33.2 wt % of TS
0.92 kg
@ 66.8 wt % of TS
1.86
41.3 %
2.78 kg
Protein @ 36.9 wt % of VS
1.17 kg
Carbohydrate @ 23.0 wt % of VS
0.73
Lipids @ 31.7 wt % of VS
1.00
ΣCPL*
2.90 kg
(91.6 wt % of VS)
Temp: 55°C
Loading:
2.1 kg VS/m³-d
HRT: 15.0 days
@ 29.2 wt % of VS
0.54 kg
53.5 %
@ 29.3 wt % of VS
0.54
25.1
@ 17.2 wt % of VS
0.32
68.2
1.40 kg
51.5 %
(75.7 wt % of VS)
VA @ 4.6 wt % of VS
0.15 kg
@ 7.1 wt % of VS
0.13 kg
13.3 %
Other organics (by diff)
0.12 kg
(3.8 wt % of VS)
0.33 kg
(175.0%)
(17.7 wt % of VS)

FS balance: effluent/feed = 93%
VS balance: effluent/feed = 104%
Carbon recovery in gas = 37%
Energy recovery in gas = 39%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

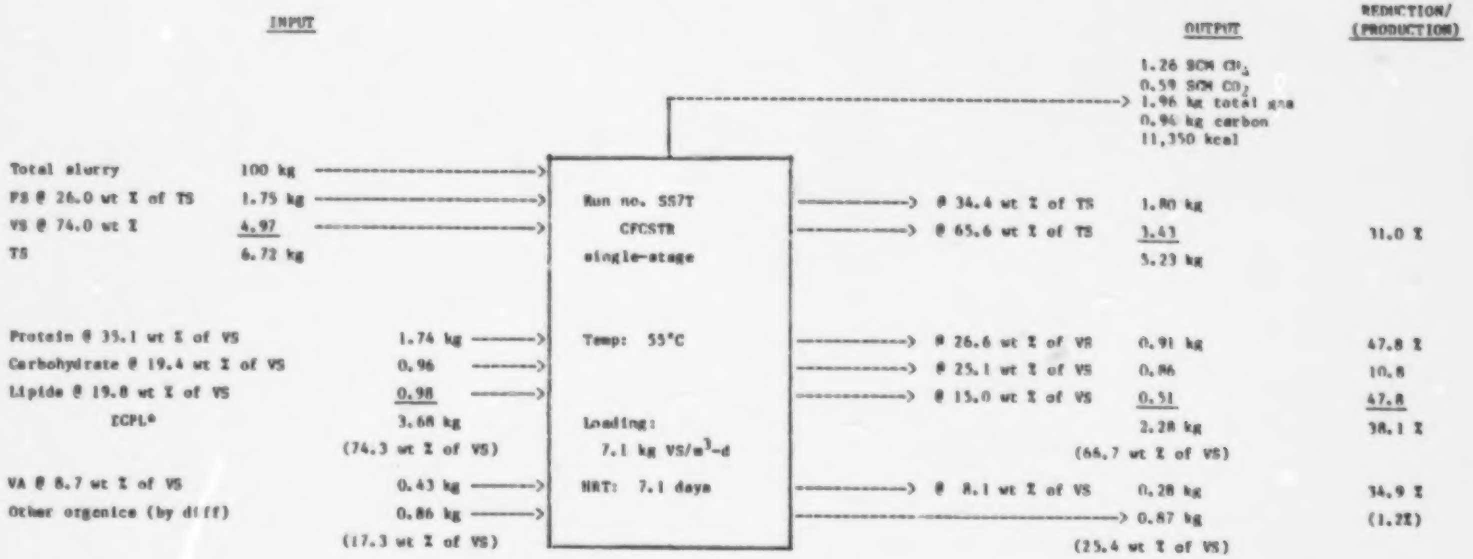
Figure B-4. Mass balances for single-stage CFCSTR thermophilic Run SS15T conducted with Hanover Park sludge at a 15-day HRT

TABLE B-9. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. SS7T: CFCSTR SINGLE-STAGE MESOPHILIC (55°C) DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
7/27/84	0.04	31.52	0.00	68.43
7/29/84	0.10	31.43	0.17	68.31
8/3/84	0.07	32.24	0.00	67.69

TABLE B-10. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. SS7T CFCSTR SINGLE-STAGE MESOPHILIC (55°C) DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
7/27/84	145	1,530	122	0	572	0	47	1,829	0
7/29/84	135	1,876	69	0	653	0	64	2,121	0
8/3/84	352	1,718	298	0	648	0	70	2,365	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
Total slurry 100 kg
FS @ 26.0 wt % of TS 1.75 kg
VS @ 74.0 wt % 4.97
TS 6.72 kg
Protein @ 35.1 wt % of VS 1.74 kg
Carbohydrate @ 19.4 wt % of VS 0.96
Lipids @ 19.8 wt % of VS 0.98
ΣCPL* 3.68 kg
(74.3 wt % of VS)
VA @ 8.7 wt % of VS 0.43 kg
Other organics (by diff) 0.86 kg
(17.3 wt % of VS)
Run no. SS7T
CFCSTR
single-stage
Temp: 55°C
Loading:
7.1 kg VS/m³-d
HRT: 7.1 days
1.26 SCM CH₄
0.59 SCM CO₂
1.96 kg total gas
0.94 kg carbon
11,350 kcal
@ 34.4 wt % of TS 1.80 kg
@ 65.6 wt % of TS 3.43 31.0 %
5.23 kg
@ 26.6 wt % of VS 0.91 kg 47.8 %
@ 25.1 wt % of VS 0.86 10.8
@ 15.0 wt % of VS 0.51 47.8
2.28 kg 38.1 %
(66.7 wt % of VS)
@ 8.1 wt % of VS 0.28 kg 34.9 %
0.87 kg (1.2%)
(25.4 wt % of VS)

FS balance: effluent/feed = 103%
VS balance: effluent/feed = 108%
Carbon recovery in gas = 32%
Energy recovery in gas = 35%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

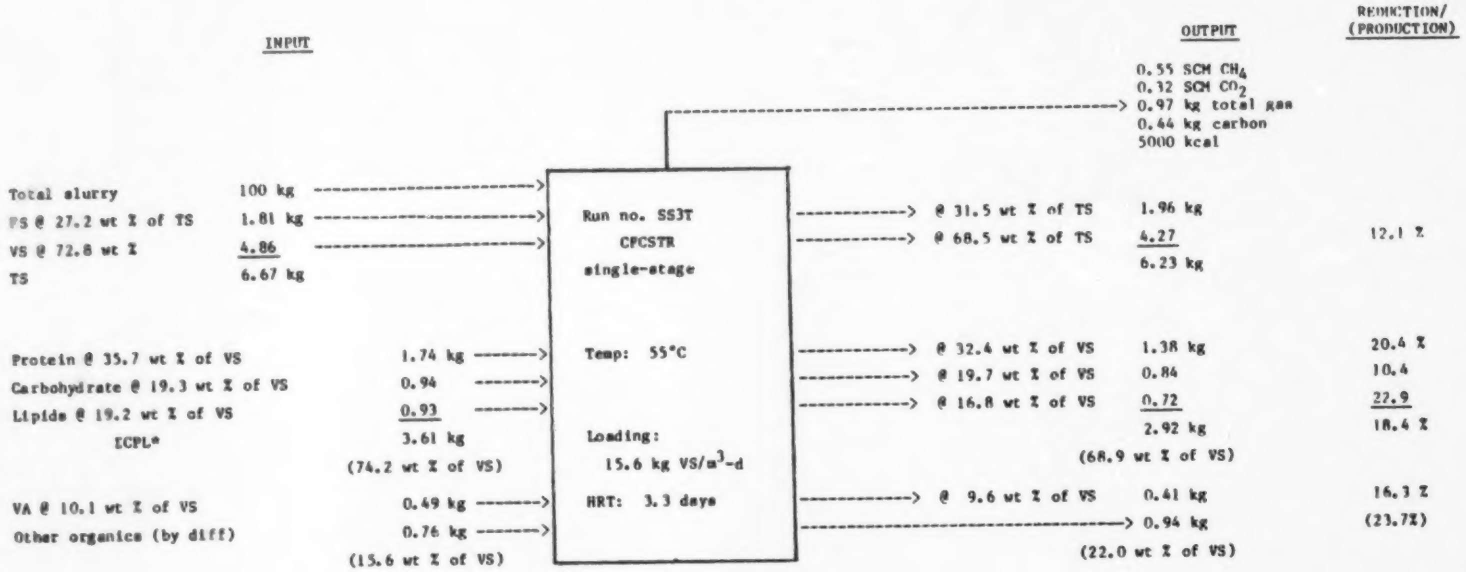
Figure B-5. Mass balances for single-stage CFCSTR thermophilic Run SS7T conducted with Hanover Park sludge at a 7-day HRT

TABLE B-11. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. SS3T: CFCSTR SINGLE-STAGE MESOPHILIC (55°C) DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 3-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
8/7/84	0.00	36.82	0.35	62.83
8/11/84	0.00	37.54	0.38	62.09
8/16/84	--	35.28	0.36	64.37
8/17/84	--	33.62	0.28	66.10

TABLE B-12. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. SS3T: CFCSTR SINGLE-STAGE MESOPHILIC (55°C) DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 3-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
8/7/84	937	1,324	473	390	863	0	126	3,172	0
8/10/84	1,128	1,496	461	338	890	0	131	3,476	0
8/15/84	1,052	1,373	300	299	772	126	148	3,178	0
8/17/84	1,064	1,322	267	258	717	0	148	2,992	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.55 SCM CH_4
0.32 SCM CO_2
0.97 kg total gas
0.44 kg carbon
5000 kcal
Total slurry 100 kg
FS @ 27.2 wt % of TS 1.81 kg
VS @ 72.8 wt % 4.86
TS 6.67 kg
Run no. SS3T
CFCSTR
single-stage
@ 31.5 wt % of TS 1.96 kg
@ 68.5 wt % of TS 4.27
6.23 kg
12.1 %
Protein @ 35.7 wt % of VS 1.74 kg
Carbohydrate @ 19.3 wt % of VS 0.94
Lipids @ 19.2 wt % of VS 0.93
ΣCPL* 3.61 kg
(74.2 wt % of VS)
Temp: 55°C
Loading:
15.6 kg VS/m^3-d
HRT: 3.3 days
@ 32.4 wt % of VS 1.38 kg 20.4 %
@ 19.7 wt % of VS 0.84 10.4
@ 16.8 wt % of VS 0.72 22.9
2.92 kg 18.4 %
(68.9 wt % of VS)
VA @ 10.1 wt % of VS 0.49 kg
Other organics (by diff) 0.76 kg
(15.6 wt % of VS)
@ 9.6 wt % of VS 0.41 kg 16.3 %
0.94 kg (23.7%)
(22.0 wt % of VS)

FS balance: effluent/feed = 108%

VS balance: effluent/feed = 108%

Carbon recovery in gas = 15%

Energy recovery in gas = 16%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure B-6. Mass balances for single-stage CFCSTR thermophilic Run SS3T conducted with Hanover Park sludge at a 3-day HRT

TABLE B-13. VOLATILE SOLIDS AND ORGANIC COMPONENT CONCENTRATIONS AND REDUCTIONS OBSERVED DURING STEADY-STATE MESOPHILIC (35°C) AND THERMOPHILIC (55°C) CFCSTR SINGLE-STAGE DIGESTION OF HANOVER PARK SEWAGE SLUDGE*

Run	Sample	Sample date(s)[b]	Lot	Batch	TS	VS		Crude protein		Carbohydrates		Lipids		Organic reductions, %		
					mg/L	mg/L	wt % of TS	mg/L	wt % of VS	mg/L	wt % of VS	mg/L	wt % of VS	Protein	Carbohydrate	Lipids
SS15M	Feed	1/6/83	1	3	45,100	33,500	74.28	12,315	36.76	6532	19.50	7239	21.61			
		1/6/83	1	3	45,100	33,500	74.28	--	--	--	--	9288	27.73			
		1/6/83	1	3	45,100	33,500	74.28	--	--	--	--	6700	20.00			
			Run means[c]		45,100	33,500	74.28		36.76		19.50		23.11			
			Feed means[d]						36.78		19.50		23.11			
			Final means[e]					12,315	36.76	6532	19.50	7742	23.11			
	Effluent	2/11/83	1	3	35,280	22,780	64.57	--	--	--	--	--	--			
		2/12-2/15/83	1	3	--	--	--	8781	38.55	4769	20.94	5078	22.29			
		2/12-2/15/83	1	3	--	--	--	--	--	4840	21.25	--	--			
		2/13/83	1	3	34,770	22,120	63.62	--	--	--	--	--	--			
		2/16-2/19/83	1	3	--	--	--	8938	40.41	4710	21.29	6490	29.34			
		2/16-2/19/83	1	3	--	--	--	9068	40.99	--	--	--	--			
					35,025	22,450	64.10	8976	39.98	4750	21.16	5797	25.82	27.11	27.28	25.12
SS7M	Feed	12/12-12/14/83	6	2	75,700	49,790	65.67	--	--	9958	20.02	--	--			
		12/31-1/9/84	6	2	80,360	53,580	66.69	14,669	27.38	9036	16.86	18,929	35.33			
			Run means		78,030	51,685	66.18		27.38		18.44		35.53			
			Feed means						27.30		18.44		37.98			
			Final means					14,125	27.34	9527	18.44	18,992	36.76			
	Effluent	12/12-12/14/83	6	2	54,930	32,510	59.18	--	--	6831	21.01	--	--			
		12/31-1/9/84	6	2	73,770	46,660	63.25	12,288	26.34	6736	14.44	13,426	28.77			
					64,350	39,585	61.52	10,427	26.34	7015	17.72	11,384	28.77	26.18	26.30	40.03
SS3M	Feed	3/28-4/6/84	8	5	63,020	47,960	76.07	16,156	34.12	12,012	25.06	12,392	25.85			
		3/28-4/6/84	8	5	63,020	47,960	76.07	--	--	11,358	23.69	--	--			
			Run means		63,020	47,960	76.07		34.12		24.38		25.85			
			Feed means						35.00		26.32		25.98			
			Final means					16,568	34.56	12,151	25.35	12,455	25.98			
	Effluent	3/28-4/6/84	8	5	55,240	40,480	73.28	12,644	31.24	6527	16.12	10,397	25.68			
		3/28-4/6/84	8	5	55,240	40,480	73.28	--	--	6978	17.24	--	--			
					55,240	40,480	73.28	12,644	31.24	6753	16.68	10,397	25.68	23.68	44.44	16.32

(continued)

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TABLE B-13 (continued)

Run	Sample	Sample date(s)[b]	Lot	Batch	TS	VS		Crude protein		Carbohydrates		Lipids		Organic reductions, %		
					mg/L	mg/L	wt % of TS	mg/L	wt % of VS	mg/L	wt % of VS	mg/L	wt % of VS	Protein	Carbohydrate	Lipids
SS15T	Feed	5/31-6/9/84	12	1	41,560	31,630	76.11	11,669	36.89	7292	23.05	10,030	31.71			
			Run means		41,560	31,630	76.11		36.89		23.05		31.71			
			Feed means						36.89		23.05		31.71			
			Final means					11,669	36.89	7292	23.05	10,030	31.71			
	Effluent	5/31-6/9/84	12	1	27,780	18,560	66.81	5425	29.23	5444	29.33	3185	17.16			
					27,780	18,560	66.81	5425	29.23	5444	29.33	3185	17.16	53.51	25.34	68.25
SS7T	Feed	8/2-8/6/84	16	1	67,250	49,740	73.96	17,188	34.56	9292	18.68	9705	19.51			
		8/2-8/6/84	16	1	67,250	49,740	73.96	--		10,238	20.58	--	--			
			Run means		67,250	49,740	73.96		34.56		19.63		19.51			
			Feed means						35.71		19.27		20.00			
			Final means					17,479	35.14	9674	19.45	9829	19.76			
	Effluent	8/2-8/8/84	16	1	52,320	34,310	65.58	9125	26.60	8856	25.81	5134	14.96			
		8/2-8/8/84	16	1	52,320	34,310	65.58	--	--	8394	24.47	--	--			
					52,320	34,310	65.58	9125	26.60	8625	25.14	5134	14.96	47.79	10.84	47.77
SS3T	Feed	8/8-8/17/84	16	1	66,735	48,615	72.85	17,369	35.73	10,363	21.32	8952	18.41			
		8/8-8/17/84	16	1	66,735	48,615	72.85	--	--	8479	17.48	--	--			
			Run means		66,735	48,615	72.85		35.73		19.40		18.41			
			Feed means						35.71		19.27		20.00			
			Final means					17,365	35.72	9402	19.34	9334	19.20			
	Effluent	8/8-8/17/84	16	1	62,340	42,705	68.50	13,825	32.37	8428	19.74	7195	16.85			
					62,340	42,705	68.50	13,825	32.37	8428	19.74	7195	16.85	20.39	10.36	22.92

---

[a] Data reported are the averages of duplicate or triplicate determinations.

[b] A single sample date indicates that the analyses were conducted on a grab sample collected that day. A time period under this column indicates the start and end dates of collection of a grab or time-composite sample used for the analyses.

[c] Run means are the averages of the feed analyses conducted for a particular steady-state run on a single feed lot and batch.

[d] Feed means are the average organic contents (expressed as weight percent of VS) of all steady-state samples collected for a particular feed lot and batch.

[e] Final means are the average feed slurry organic concentrations and contents used to determine the organic reductions. The organic contents are the average of the feed and run organic contents. Organic feed concentrations were calculated as the product of the final mean organic feed contents and the average feed volatile solids concentration for the run.

APPENDIX C

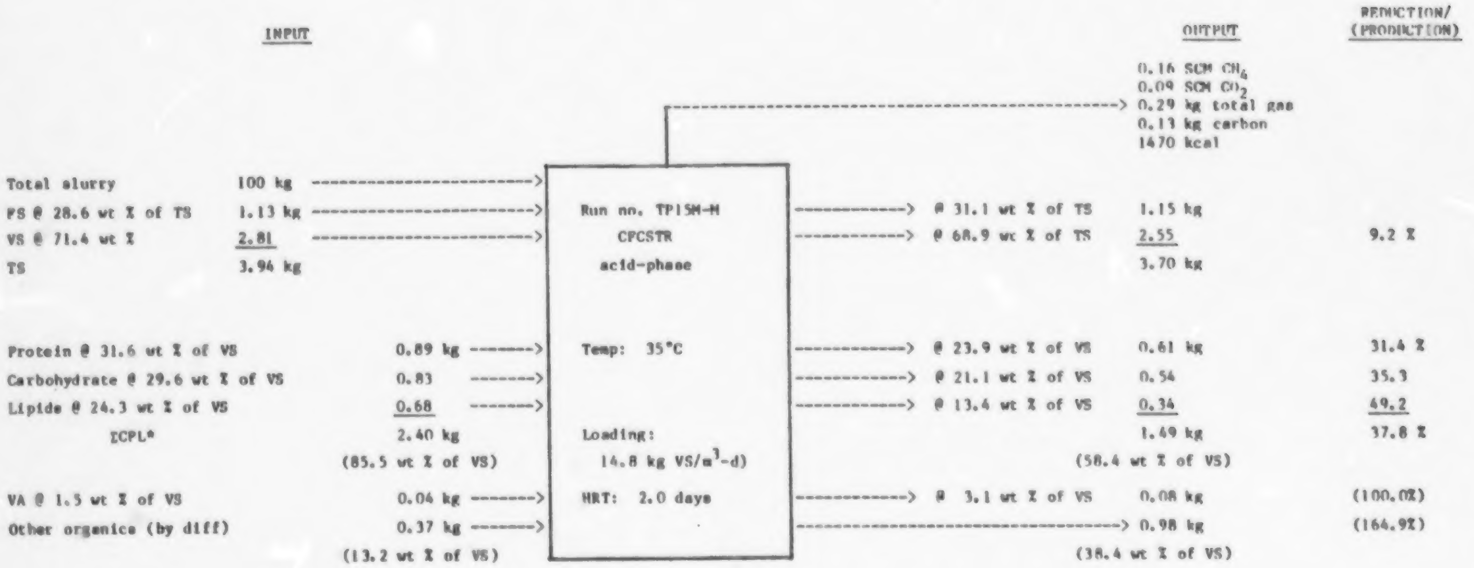
EFFLUENT ANALYSES FOR TWO-PHASE CFCSTR DIGESTION STUDIES

TABLE C-1. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. TP15M-M: CFCSTR MESO-MESO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 15-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Digester No. 332)				
10/9/83	--	38.24	1.56	60.21
10/16/83	--	39.25	0.98	59.77
10/22/83	--	37.04	1.25	61.72
10/29/83	--	36.05	0.81	63.14
11/6/83	--	35.03	1.51	63.46
11/13/83	--	33.78	0.74	65.49
11/14/83	--	35.09	2.89	62.02
11/20/83	--	34.78	0.95	64.26
Methane-phase (Digester No. 333)				
10/9/83	--	29.45	1.26	69.28
10/16/83	--	28.80	1.43	69.77
10/22/83	--	29.16	0.60	70.25
10/29/83	--	28.77	0.42	70.81
11/6/83	--	28.79	0.00	71.21
11/13/83	--	29.12	0.00	70.88
11/14/83	--	28.88	0.00	71.12
11/20/83	--	28.60	0.00	71.40

TABLE C-2. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. TP15M-M: CFCSTR MESO-MESO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 15-DAY HRT

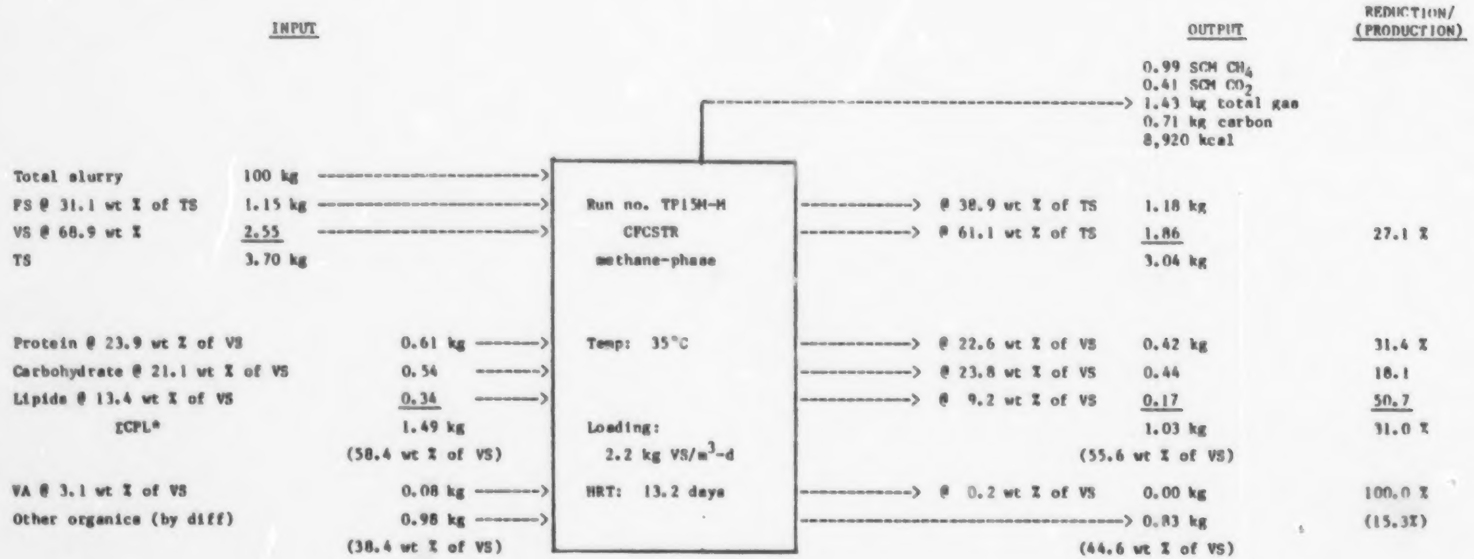
Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Digester No. 332)									
10/17/83	324	340	49	32	91	14	0	716	0
10/21/83	464	468	66	62	119	24	0	1,014	12
10/29/83	396	402	57	48	111	20	0	871	0
11/7/83	282	331	35	7	79	15	0	635	0
11/14/83	201	258	24	0	65	4	0	467	--
11/15/83	212	219	28	4	71	9	0	458	--
11/21/83	188	275	23	16	51	18	0	478	--
Methane-phase (Digester No. 333)									
10/17/83	6	3	0	0	0	0	0	8	0
10/21/83	51	13	0	0	0	0	0	61	15
10/29/83	23	0	0	0	0	0	0	23	0
11/7/83	37	30	0	0	0	0	0	62	0
11/14/83	0	0	0	0	0	0	0	0	--
11/15/83	1	0	0	0	0	0	0	1	--


INPUT
OUTPUT
REDUCTION/
(PRODUCTION)
0.16 SCM CH₄
0.09 SCM CO₂
0.29 kg total gas
0.13 kg carbon
1470 kcal
Total slurry 100 kg
FS @ 28.6 wt % of TS 1.13 kg
VS @ 71.4 wt % 2.81
TS 3.94 kg
Run no. TP15M-M
CFCSTR
acid-phase
@ 31.1 wt % of TS 1.15 kg
@ 68.9 wt % of TS 2.55 9.2 %
3.70 kg
Protein @ 31.6 wt % of VS 0.89 kg
Carbohydrate @ 29.6 wt % of VS 0.83
Lipids @ 24.3 wt % of VS 0.68
ΣCPL* 2.40 kg
(85.5 wt % of VS)
Temp: 35°C
Loading:
14.8 kg VS/m³-d)
HRT: 2.0 days
@ 23.9 wt % of VS 0.61 kg 31.4 %
@ 21.1 wt % of VS 0.54 35.3
@ 13.4 wt % of VS 0.34 49.2
1.49 kg 37.8 %
(58.4 wt % of VS)
VA @ 1.5 wt % of VS 0.04 kg
Other organics (by diff) 0.37 kg
(13.2 wt % of VS)
@ 3.1 wt % of VS 0.08 kg (100.0%)
0.98 kg (164.9%)
(38.4 wt % of VS)

FS balance: effluent/feed = 102%
VS balance: effluent/feed = 101%
Carbon recovery in gas = 8%
Energy recovery in gas = 8%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

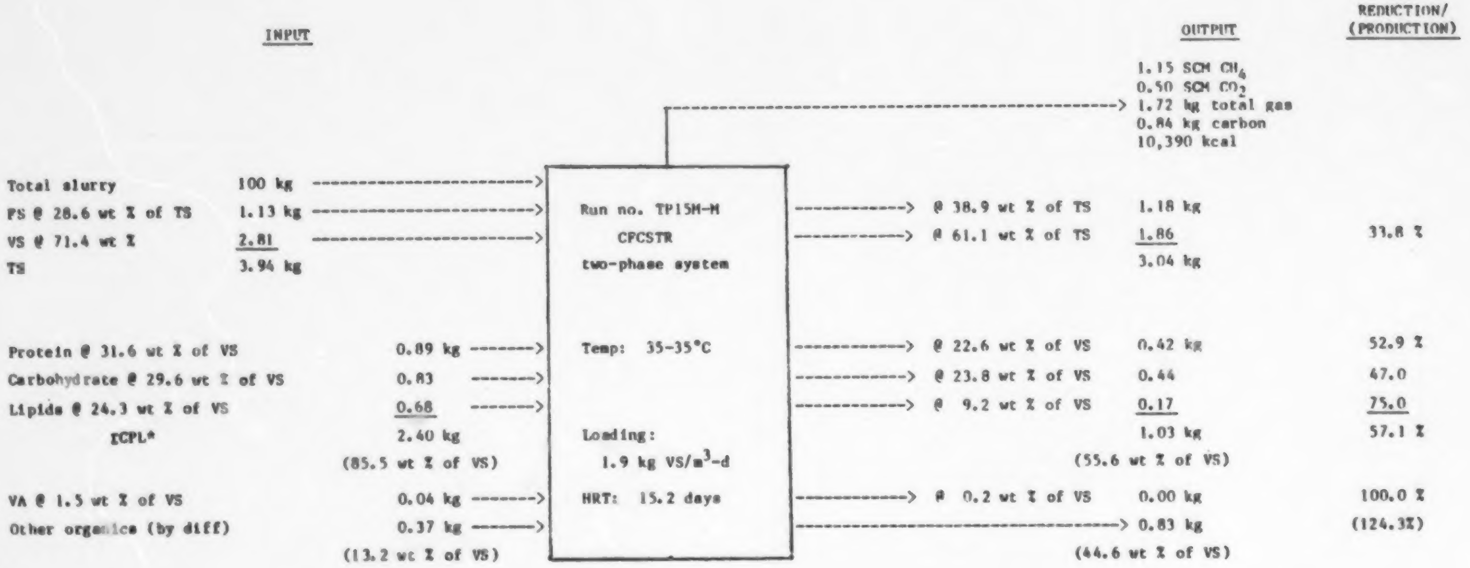
Figure C-1. Mass balances for mesophilic CFCSTR acid-phase digester for meso-meso CFCSTR two-phase Run TP15M-M conducted with Hanover Park sludge at a 15-day HRT


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.99 SCM CH_4
0.41 SCM CO_2
1.43 kg total gas
0.71 kg carbon
8,920 kcal
Total slurry
100 kg
FS @ 31.1 wt % of TS
1.15 kg
VS @ 68.9 wt %
2.55
TS
3.70 kg
Run no. TP15M-M
CFCSTR
methane-phase
@ 38.9 wt % of TS
1.18 kg
@ 61.1 wt % of TS
1.86
27.1 %
3.04 kg
Protein @ 23.9 wt % of VS
0.61 kg
Carbohydrate @ 21.1 wt % of VS
0.54
Lipids @ 13.4 wt % of VS
0.34
ΣCPL*
1.49 kg
(58.4 wt % of VS)
Temp: 35°C
Loading:
2.2 kg VS/m^3-d
HRT: 13.2 days
@ 22.6 wt % of VS
0.42 kg
31.4 %
@ 23.8 wt % of VS
0.44
18.1
@ 9.2 wt % of VS
0.17
50.7
1.03 kg
31.0 %
(55.6 wt % of VS)
VA @ 3.1 wt % of VS
0.08 kg
@ 0.2 wt % of VS
0.00 kg
100.0 %
Other organics (by diff)
0.98 kg
0.83 kg
(15.3%)
(38.4 wt % of VS)
(44.6 wt % of VS)

FS balance: effluent/feed = 103%
VS balance: effluent/feed = 129%
Carbon recovery in gas = 48%
Energy recovery in gas = 54%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure C-2. Mass balances for mesophilic CFCSTR methane-phase digester for meso-meso CFCSTR two-phase Run TP15M-M conducted with Hanover Park sludge at a 15-day system HRT


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
1.15 SCM CH4
0.50 SCM CO2
1.72 kg total gas
0.84 kg carbon
10,390 kcal
Total slurry 100 kg
FS @ 28.6 wt % of TS 1.13 kg
VS @ 71.4 wt % 2.81
TS 3.94 kg
Run no. TP15M-M
CFCSTR
two-phase system
@ 38.9 wt % of TS 1.18 kg
@ 61.1 wt % of TS 1.86
3.04 kg
33.8 %
Protein @ 31.6 wt % of VS 0.89 kg
Carbohydrate @ 29.6 wt % of VS 0.83
Lipids @ 24.3 wt % of VS 0.68
ΣCPL* 2.40 kg
(85.5 wt % of VS)
Temp: 35-35°C
Loading:
1.9 kg VS/m3-d
HRT: 15.2 days
@ 22.6 wt % of VS 0.42 kg 52.9 %
@ 23.8 wt % of VS 0.44 47.0
@ 9.2 wt % of VS 0.17 75.0
1.03 kg 57.1 %
(55.6 wt % of VS)
VA @ 1.5 wt % of VS 0.04 kg
Other organics (by diff) 0.37 kg
(13.2 wt % of VS)
@ 0.2 wt % of VS 0.00 kg 100.0 %
0.83 kg (124.3%)
(44.6 wt % of VS)

FS balance: effluent/feed = 104%
VS balance: effluent/feed = 127%
Carbon recovery in gas = 51%
Energy recovery in gas = 57%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

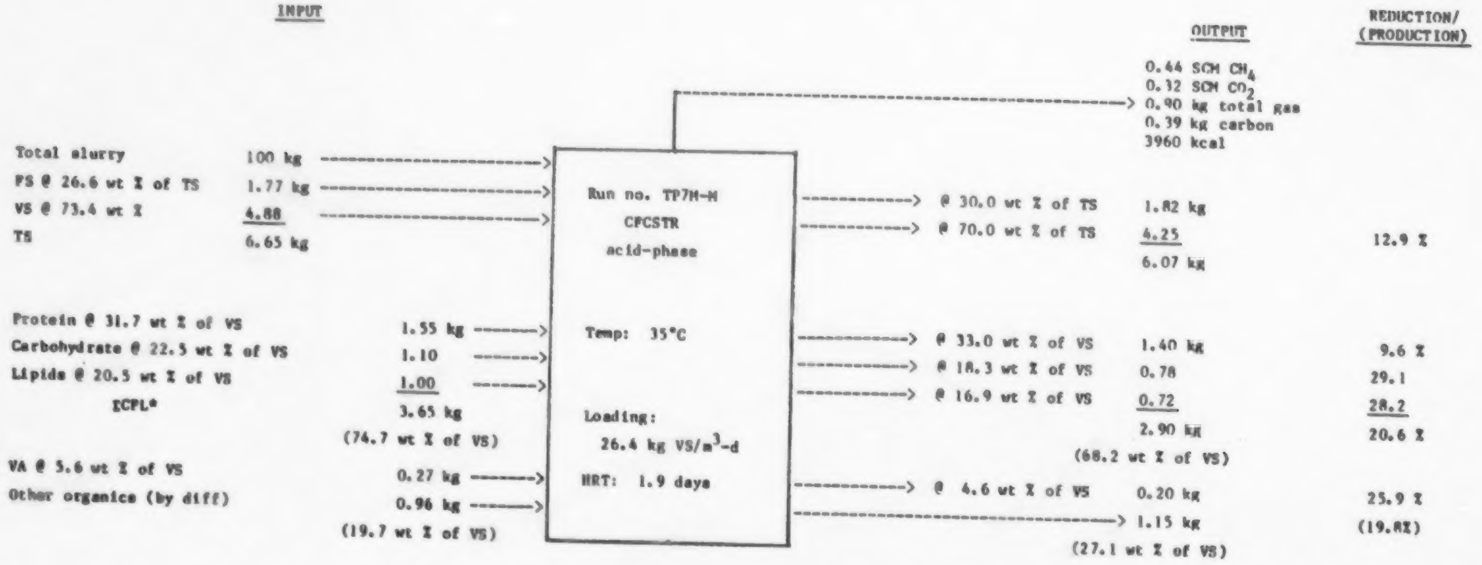
Figure C-3. Mass balances for meso-meso CFCSTR two-phase Run TP15M-M conducted with Hanover Park sludge at a 15-day system HRT

TABLE C-3. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. TP7M-M: CFCSTR MESO-MESO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Digester No. 334)				
9/21/84	--	42.25	0.12	57.63
9/24/83	0.00	43.07	0.67	56.26
9/26/84	--	42.53	0.33	57.14
9/28/84	--	40.94	0.42	58.65
Methane-phase (Digester No. 333)				
9/21/84	--	31.56	0.14	68.31
9/24/84	--	31.51	0.00	68.49
9/26/84	--	32.00	0.05	67.95
9/28/84	--	32.25	0.42	67.32

TABLE C-4. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. TP7M-M: CFCSTR MESO-MESO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Iso-butyric, mg/L	Butyric, mg/L	Iso-valeric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Digester No. 334)									
9/21/84	703	701	87	136	103	42	68	1,544	0
9/24/84	861	753	113	214	137	38	55	1,826	0
9/26/84	845	764	116	207	149	58	2	1,808	0
9/28/84	475	693	121	82	155	107	0	1,329	0
Methane-phase (Digester No. 333)									
9/21/84	81	11	0	0	0	6	62	125	0
9/24/84	99	9	0	0	0	32	44	148	0
9/26/84	72	47	0	0	0	2	26	124	0
9/28/84	1	47	0	0	0	0	0	39	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.44 SCM CH4
0.32 SCM CO2
0.90 kg total gas
0.39 kg carbon
3960 kcal
Total slurry 100 kg
FS @ 26.6 wt % of TS 1.77 kg
VS @ 73.4 wt % 4.88
TS 6.65 kg
Run no. TP7M-M
CFCSTR
acid-phase
@ 30.0 wt % of TS 1.82 kg
@ 70.0 wt % of TS 4.25
6.07 kg
12.9 %
Protein @ 31.7 wt % of VS 1.55 kg
Carbohydrate @ 22.5 wt % of VS 1.10
Lipids @ 20.5 wt % of VS 1.00
ΣCPL* 3.65 kg
(74.7 wt % of VS)
Temp: 35°C
@ 33.0 wt % of VS 1.40 kg 9.6 %
@ 18.3 wt % of VS 0.78 29.1
@ 16.9 wt % of VS 0.72 28.2
2.90 kg 20.6 %
(68.2 wt % of VS)
Loading:
26.4 kg VS/m3-d
HRT: 1.9 days
VA @ 5.6 wt % of VS 0.27 kg
Other organics (by diff) 0.96 kg
(19.7 wt % of VS)
@ 4.6 wt % of VS 0.20 kg 25.9 %
1.15 kg (19.8%)
(27.1 wt % of VS)

FS balance: effluent/feed = 103%

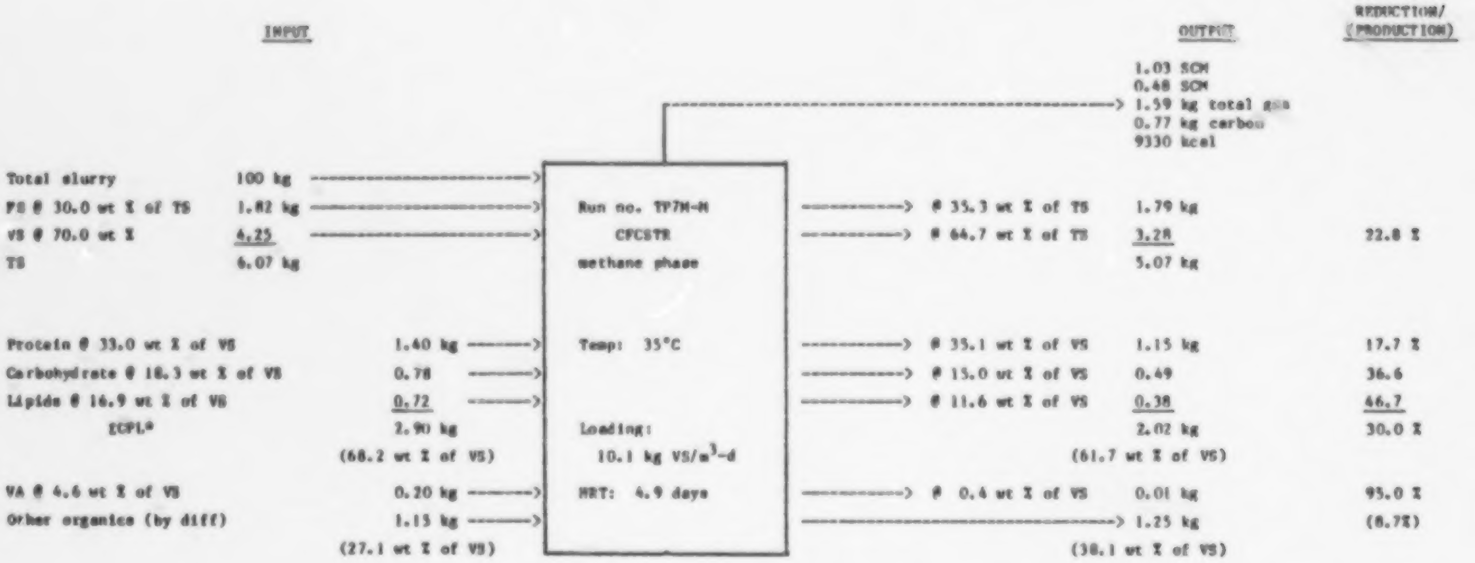
VS balance: effluent/feed = 106%

Carbon recovery in gas = 14%

Energy recovery in gas = 13%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

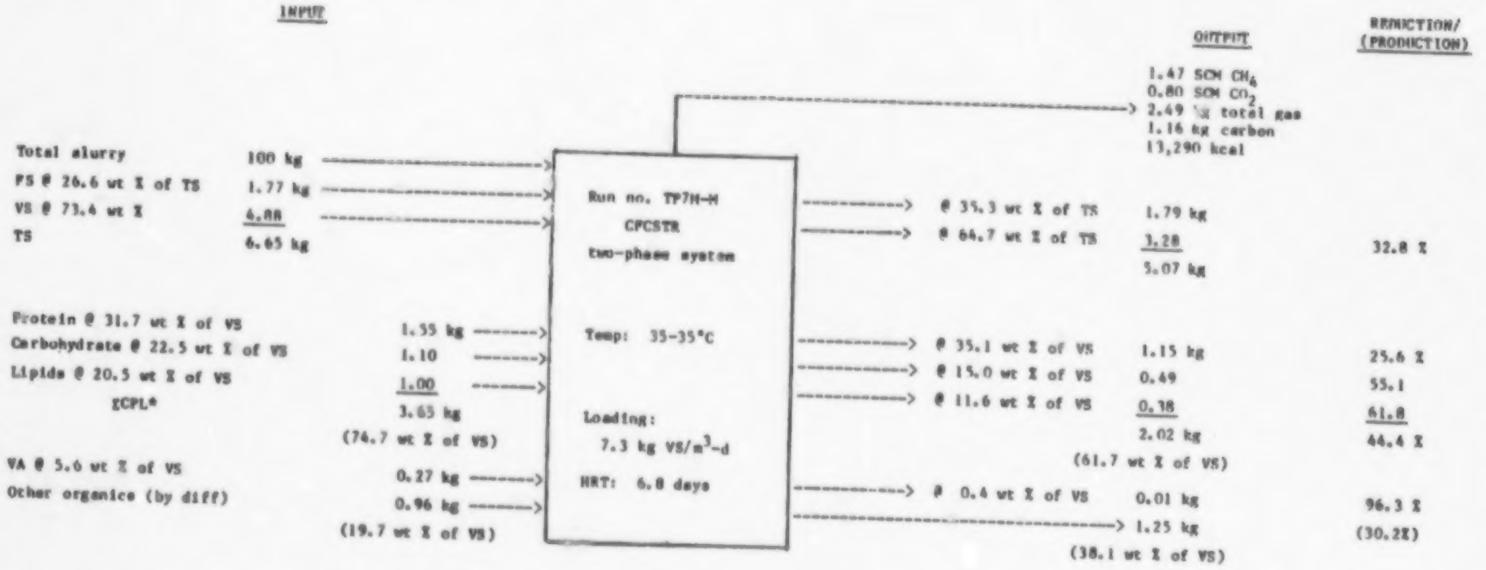
Figure C-4. Mass balances for mesophilic CFCSTR acid-phase digester for meso-meso CFCSTR two-phase Run TP7M-M conducted with Hanover Park sludge at a 7-day system HRT


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
1.03 SCM
0.48 SCM
1.59 kg total gas
0.77 kg carbon
9330 kcal
Total slurry 100 kg
FS @ 30.0 wt % of TS 1.82 kg
VS @ 70.0 wt % 4.25
TS 6.07 kg
Run no. TP7M-M
CFCSTR
methane phase
@ 35.3 wt % of TS 1.79 kg
@ 64.7 wt % of TS 3.28 22.8 %
5.07 kg
Protein @ 33.0 wt % of VS 1.40 kg
Carbohydrate @ 18.3 wt % of VS 0.78
Lipids @ 16.9 wt % of VS 0.72
ΣCPL* 2.90 kg
(68.2 wt % of VS)
Temp: 35°C
Loading:
10.1 kg VS/m3-d
HRT: 4.9 days
@ 35.1 wt % of VS 1.15 kg 17.7 %
@ 15.0 wt % of VS 0.49 36.6
@ 11.6 wt % of VS 0.38 46.7
2.02 kg 30.0 %
(61.7 wt % of VS)
VA @ 4.6 wt % of VS 0.20 kg
Other organics (by diff) 1.15 kg
(27.1 wt % of VS)
@ 0.4 wt % of VS 0.01 kg 95.0 %
1.25 kg (8.7%)
(38.1 wt % of VS)

FS balance: effluent/feed = 98%
VS balance: effluent/feed = 115%
Carbon recovery in gas = 31%
Energy recovery in gas = 34%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure C-5. Mass balances for mesophilic CFCSTR methane-phase digester for meso-meso CFCSTR two-phase Run TP7M-M conducted with Hanover Park sludge at a 7-day system HRT


INPUT
OUTPUT
REDUCTION/
(PRODUCTION)
Total slurry
100 kg
FS @ 26.6 wt % of TS
1.77 kg
VS @ 73.4 wt %
4.88
TS
6.65 kg
Protein @ 31.7 wt % of VS
1.55 kg
Carbohydrate @ 22.5 wt % of VS
1.10
Lipids @ 20.5 wt % of VS
1.00
ΣCPL*
3.65 kg
(74.7 wt % of VS)
VA @ 5.6 wt % of VS
0.27 kg
Other organics (by diff)
0.96 kg
(19.7 wt % of VS)
Run no. TP7M-M
CFCSTR
two-phase system
Temp: 35-35°C
Loading:
7.3 kg VS/m³-d
HRT: 6.8 days
1.47 SCM CH4
0.80 SCM CO2
2.49 [illegible] total gas
1.16 kg carbon
13,290 kcal
@ 35.3 wt % of TS
1.79 kg
@ 64.7 wt % of TS
3.28
5.07 kg
32.8 %
@ 35.1 wt % of VS
1.15 kg
25.6 %
@ 15.0 wt % of VS
0.49
55.1
@ 11.6 wt % of VS
0.38
61.8
2.02 kg
44.4 %
(61.7 wt % of VS)
@ 0.4 wt % of VS
0.01 kg
96.3 %
1.25 kg
(30.2%)
(38.1 wt % of VS)

FS balance: effluent/feed = 101%
VS balance: effluent/feed = 118%
Carbon recovery in gas = 41%
Energy recovery in gas = 42%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

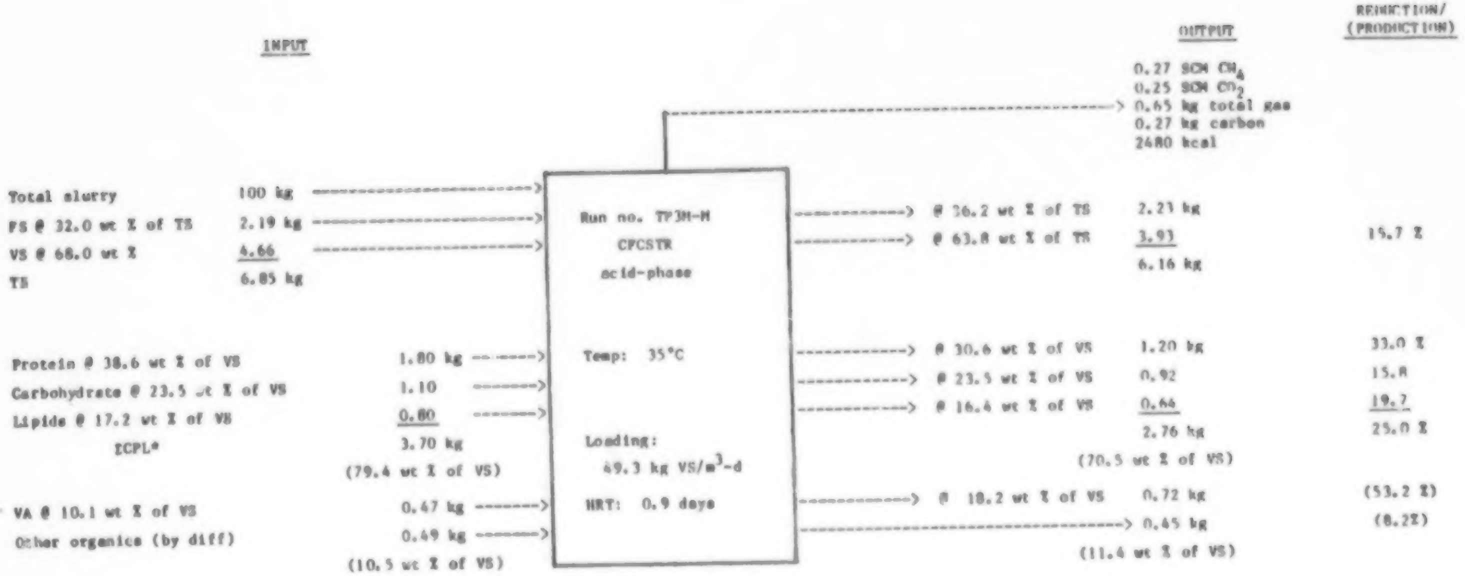
Figure C-6. Mass balances for meso-meso CFCSTR two-phase Run TP7M-M conducted with Hanover Park sludge at a 7-day system HRT

TABLE C-5. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. TP3M-M: CFCSTR MESO-MESO TWO-PHASE DIGESTION OF MIXED DOWNERS GROVE PRIMARY AND STICKNEY ACTIVATED SLUDGES CONDUCTED AT A 3-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Digester No. 334)				
12/13/84	--	47.88	0.63	51.50
12/15/84	--	48.06	1.68	50.26
12/18/84	--	45.59	0.32	54.08
12/20/84	0.05	44.48	0.29	55.18
Methane-phase (Digester No. 333)				
12/13/84	--	35.10	0.00	64.90
12/15/84	--	36.64	1.59	61.77
12/18/84	--	36.02	0.00	63.98
12/20/84	0.00	38.06	0.00	61.95

TABLE C-6. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. TP3M-M: CFCSTR MESO-MESO TWO-PHASE DIGESTION OF MIXED DOWNERS GROVE PRIMARY AND STICKNEY ACTIVATED SLUDGES CONDUCTED AT A 3-DAY HRT

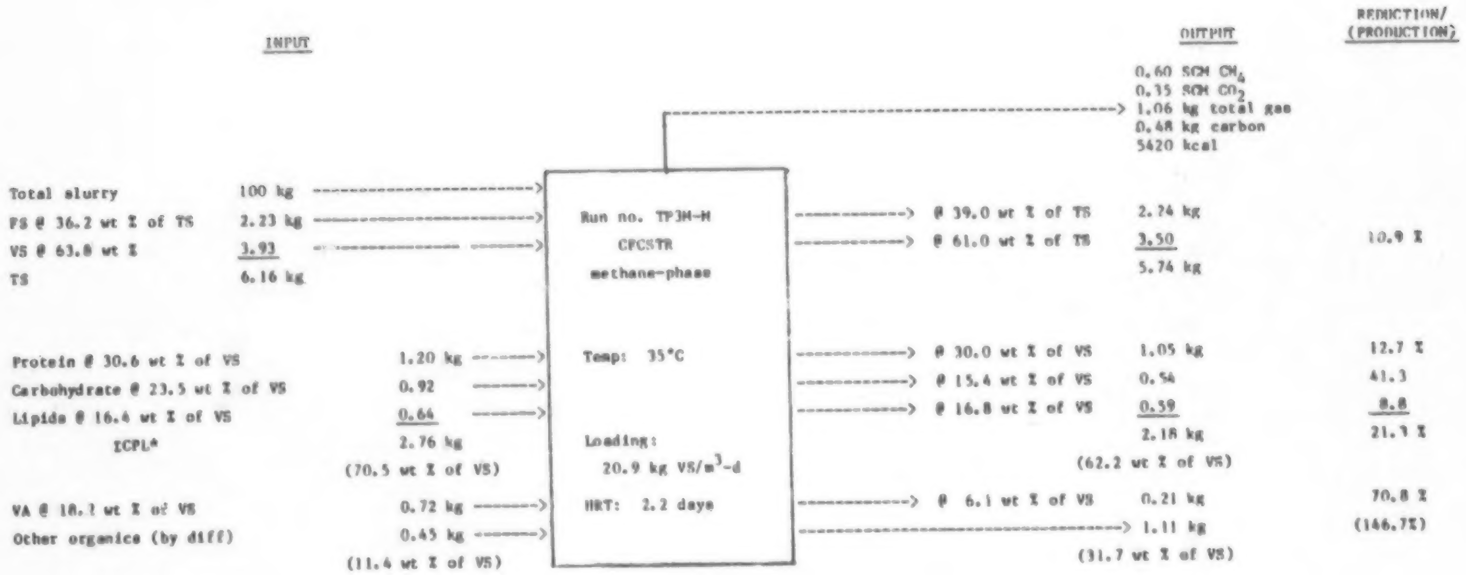
Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Digester No. 334)									
12/13/84	2,133	1,344	282	781	626	3,559	0	6,405	0
12/15/84	2,477	1,312	294	849	682	2,014	101	5,956	37
12/18/84	2,336	1,615	321	778	618	1,063	18	5,391	0
12/20/84	1,762	1,342	254	588	458	991	83	4,319	39
Methane-phase (Digester No. 333									
12/13/84	187	1,306	18	0	68	136	0	1,379	0
12/15/84	293	1,336	19	0	201	69	52	1,575	0
12/18/84	276	1,746	68	0	267	75	0	1,939	0
12/20/84	114	1,620	95	0	245	264	71	1,827	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.27 SCM CH_4
0.25 SCM CO_2
0.65 kg total gas
0.27 kg carbon
2480 kcal
Total slurry 100 kg
FS @ 32.0 wt % of TS 2.19 kg
VS @ 68.0 wt % 4.66
TS 6.85 kg
Run no. TP3M-M
CFCSTR
acid-phase
@ 36.2 wt % of TS 2.23 kg
@ 63.8 wt % of TS 3.93 15.7 %
6.16 kg
Protein @ 38.6 wt % of VS 1.80 kg
Carbohydrate @ 23.5 wt % of VS 1.10
Lipids @ 17.2 wt % of VS 0.80
ΣCPL* 3.70 kg
(79.4 wt % of VS)
Temp: 35°C
@ 30.6 wt % of VS 1.20 kg 33.0 %
@ 23.5 wt % of VS 0.92 15.8
@ 16.4 wt % of VS 0.64 19.7
2.76 kg 25.0 %
(70.5 wt % of VS)
Loading:
49.3 kg VS/m^3-d
HRT: 0.9 days
VA @ 10.1 wt % of VS 0.47 kg
Other organics (by diff) 0.49 kg
(10.5 wt % of VS)
@ 18.2 wt % of VS 0.72 kg (53.2 %)
0.45 kg (8.2%)
(11.4 wt % of VS)

FS balance: effluent/feed = 102%
VS balance: effluent/feed = 102%
Carbon recovery in gas = 10%
Energy recovery in gas = 8%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

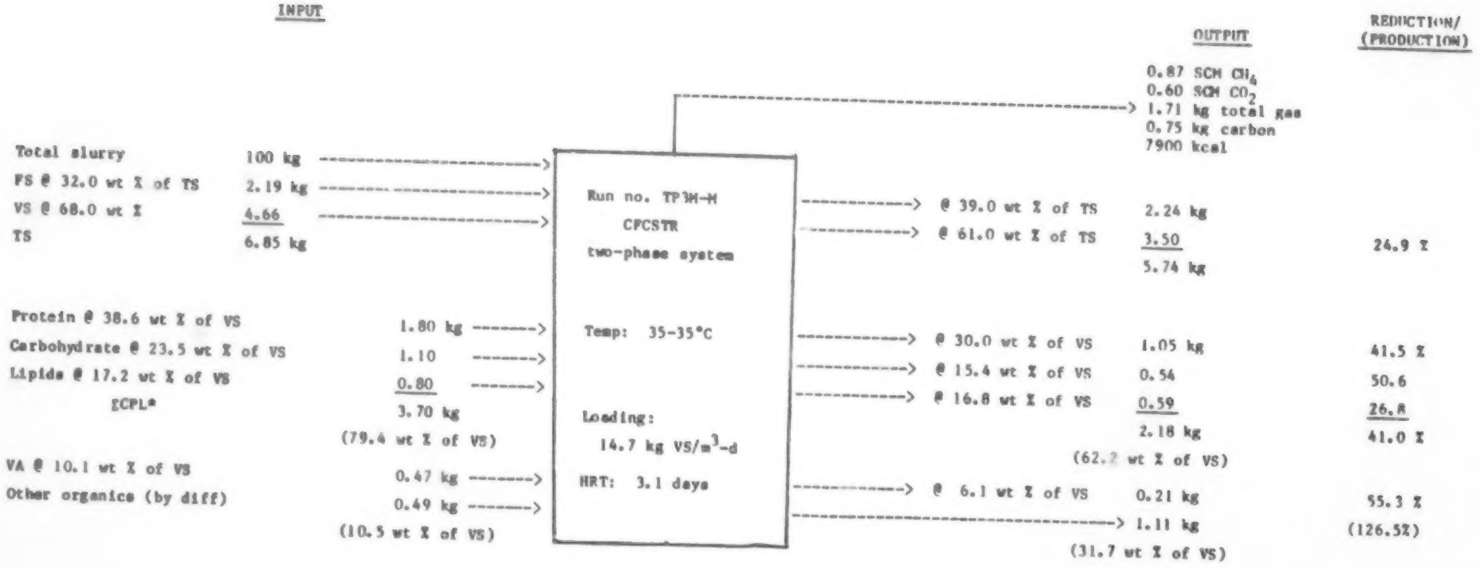
Figure C-7. Mass balances for mesophilic CFCSTR acid-phase digester for meso-meso CFCSTR two-phase Run TP3M-M conducted with mixed Downers Grove primary and Stickney activated sludges at a 3-day system HRT


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.60 SCM CH_4
0.35 SCM CO_2
1.06 kg total gas
0.48 kg carbon
5420 kcal
Total slurry 100 kg
FS @ 36.2 wt % of TS 2.23 kg
VS @ 63.8 wt % 3.93
TS 6.16 kg
Run no. TP3M-M
CFCSTR
methane-phase
@ 39.0 wt % of TS 2.24 kg
@ 61.0 wt % of TS 3.50 10.9 %
5.74 kg
Protein @ 30.6 wt % of VS 1.20 kg
Carbohydrate @ 23.5 wt % of VS 0.92
Lipids @ 16.4 wt % of VS 0.64
ΣCPL* 2.76 kg
(70.5 wt % of VS)
Temp: 35°C
Loading:
20.9 kg VS/m^3-d
HRT: 2.2 days
@ 30.0 wt % of VS 1.05 kg 12.7 %
@ 15.4 wt % of VS 0.54 41.3
@ 16.8 wt % of VS 0.59 8.8
2.18 kg 21.3 %
(62.2 wt % of VS)
VA @ 18.1 wt % of VS 0.72 kg
Other organics (by diff) 0.45 kg
(11.4 wt % of VS)
@ 6.1 wt % of VS 0.21 kg 70.8 %
1.11 kg (146.7%)
(31.7 wt % of VS)

FS balance: effluent/feed = 100%
VS balance: effluent/feed = 116%
Carbon recovery in gas = 21%
Energy recovery in gas = 21%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure C-8. Mass balances for mesophilic CFCSTR methane-phase digester for meso-meso CFCSTR two-phase Run TP3M-M conducted with mixed Downers Grove primary and Stickney activated sludges at a 3-day system HRT


INPUT
Total slurry
100 kg
FS @ 32.0 wt % of TS
2.19 kg
VS @ 68.0 wt %
4.66
TS
6.85 kg
Protein @ 38.6 wt % of VS
1.80 kg
Carbohydrate @ 23.5 wt % of VS
1.10
Lipids @ 17.2 wt % of VS
0.80
ΣCPL*
3.70 kg
(79.4 wt % of VS)
VA @ 10.1 wt % of VS
0.47 kg
Other organics (by diff)
0.49 kg
(10.5 wt % of VS)
Run no. TP3M-M
CFCSTR
two-phase system
Temp: 35-35°C
Loading:
14.7 kg VS/m³-d
HRT: 3.1 days
OUTPUT
0.87 SCM CH₄
0.60 SCM CO₂
1.71 kg total gas
0.75 kg carbon
7900 kcal
@ 39.0 wt % of TS
2.24 kg
@ 61.0 wt % of TS
3.50
5.74 kg
@ 30.0 wt % of VS
1.05 kg
@ 15.4 wt % of VS
0.54
@ 16.8 wt % of VS
0.59
2.18 kg
(62.2 wt % of VS)
@ 6.1 wt % of VS
0.21 kg
1.11 kg
(31.7 wt % of VS)
REDUCTION/
(PRODUCTION)
24.9 %
41.5 %
50.6
26.8
41.0 %
55.3 %
(126.5%)

FS balance: effluent/feed = 102%
VS balance: effluent/feed = 112%
Carbon recovery in gas = 28%
Energy recovery in gas = 26%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

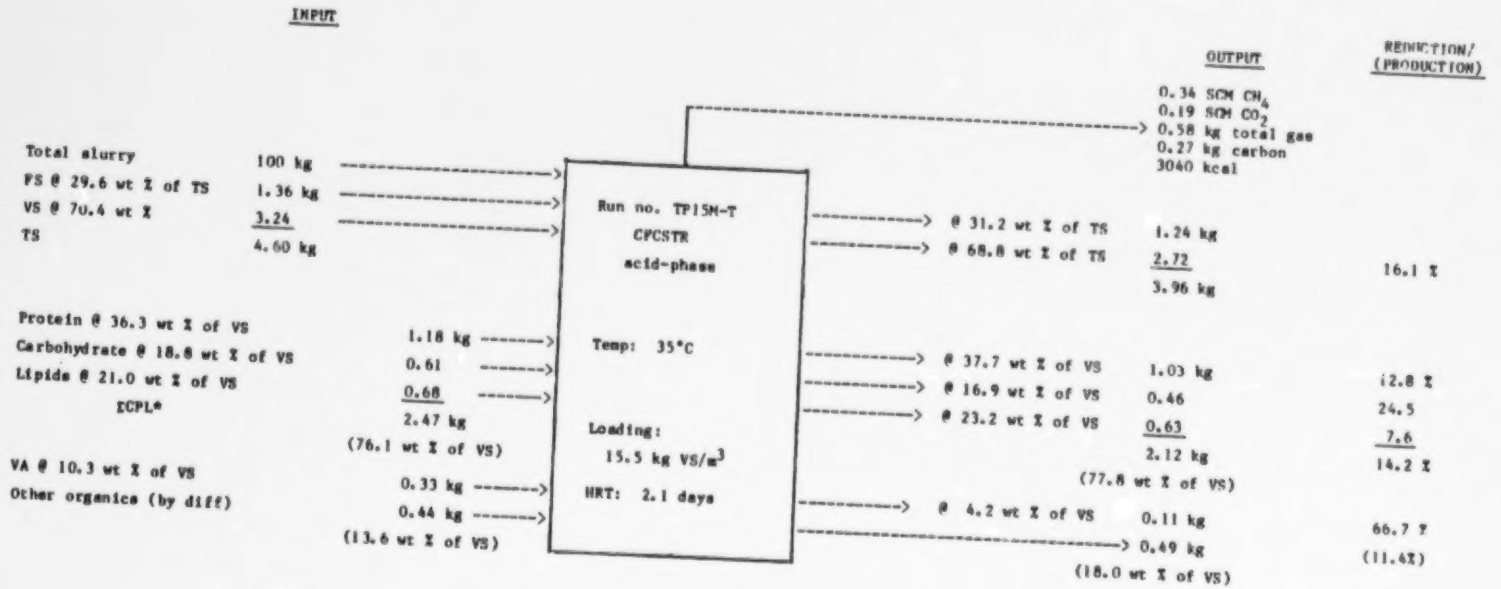
Figure C-9. Mass balances for meso-meso CFCSTR two-phase Run TP3M-M conducted with mixed Downers Grove primary and Stickney activated sludges at a 3-day system HRT

TABLE C-7. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. TP15M-T: CFCSTR MESO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 15-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Digester No. 334)				
7/20/84	--	38.83	0.42	60.75
7/27/84	--	38.43	0.36	61.21
8/3/84	0.00	33.74	0.47	65.79
8/7/84	0.00	34.03	0.30	65.67
8/11/84	0.00	34.86	0.37	64.77
8/16/84	--	33.27	0.58	66.15
Methane-phase (Digester No. 337)				
7/20/84	--	32.26	0.32	67.42
7/27/84	--	31.48	0.17	68.35
8/3/84	--	30.54	0.66	68.79
8/7/84	0.00	31.51	0.20	68.28
8/11/84	0.00	31.39	0.62	67.99
8/16/84	--	30.55	0.37	69.09

TABLE C-8. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. TP15M-T: CFCSTR MESO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 15-DAY HRT

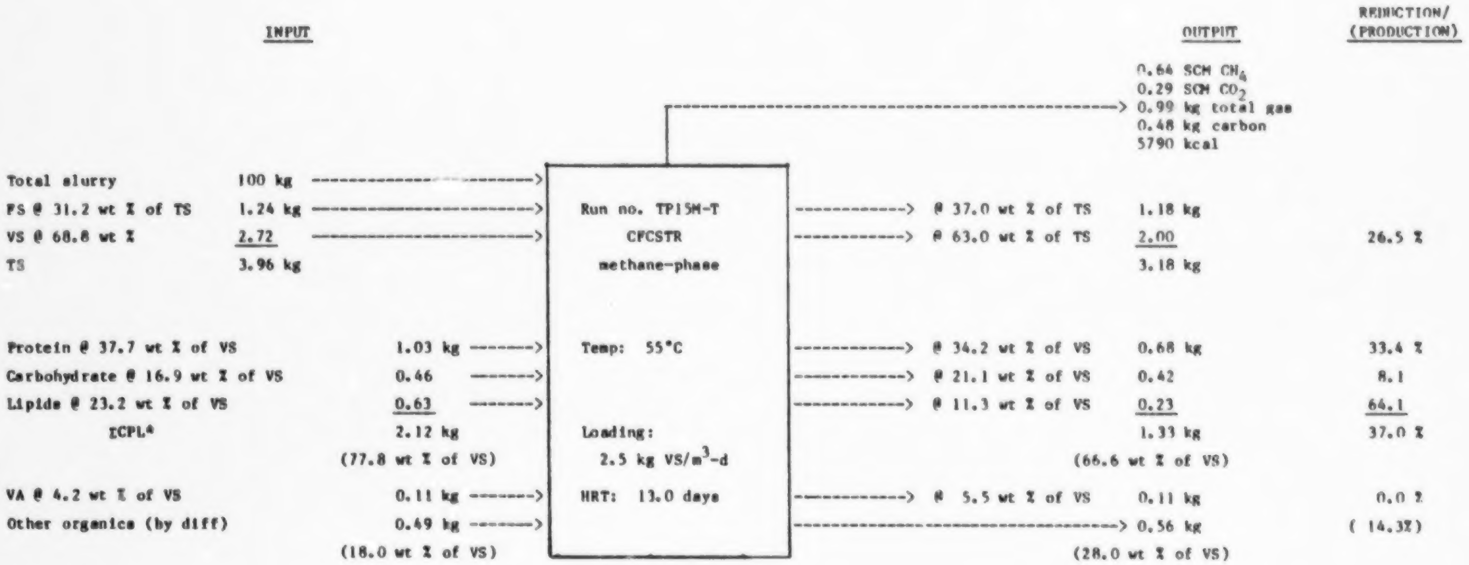
Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Digester No. 334)									
7/20/84	396	453	49	71	108	75	17	962	0
7/27/84	284	313	46	50	84	43	13	684	0
8/3/84	590	448	29	74	83	0	20	1,082	0
8/7/84	502	351	37	70	83	14	29	931	0
8/10/84	605	504	7	43	75	0	29	1,106	0
8/16/84	602	377	0	64	52	0	92	1,030	0
Methane-phase (Digester No. 337)									
7/20/84	271	836	0	0	197	0	22	1,076	0
7/27/84	213	579	17	0	143	0	27	792	0
8/3/84	122	713	106	0	231	0	34	925	0
8/7/84	119	633	68	0	191	0	23	803	0
8/10/84	203	621	0	0	185	0	24	827	0
8/16/84	143	648	0	0	165	0	25	779	0


INPUT
Total slurry
100 kg
FS @ 29.6 wt % of TS
1.36 kg
VS @ 70.4 wt %
3.24
TS
4.60 kg
Protein @ 36.3 wt % of VS
1.18 kg
Carbohydrate @ 18.8 wt % of VS
0.61
Lipids @ 21.0 wt % of VS
0.68
ΣCPL*
2.47 kg
(76.1 wt % of VS)
VA @ 10.3 wt % of VS
0.33 kg
Other organics (by diff)
0.44 kg
(13.6 wt % of VS)
Run no. TP15M-T
CFCSTR
acid-phase
Temp: 35°C
Loading:
15.5 kg VS/m^3
HRT: 2.1 days
OUTPUT
REDUCTION/
(PRODUCTION)
0.34 SCM CH$_4$
0.19 SCM CO$_2$
0.58 kg total gas
0.27 kg carbon
3040 kcal
@ 31.2 wt % of TS
1.24 kg
@ 68.8 wt % of TS
2.72
3.96 kg
16.1 %
@ 37.7 wt % of VS
1.03 kg
12.8 %
@ 16.9 wt % of VS
0.46
24.5
@ 23.2 wt % of VS
0.63
7.6
2.12 kg
14.2 %
(77.8 wt % of VS)
@ 4.2 wt % of VS
0.11 kg
66.7 %
0.49 kg
(11.4%)
(18.0 wt % of VS)

FS balance: effluent/feed = 91%
VS balance: effluent/feed = 102%
Carbon recovery in gas = 14%
Energy recovery in gas = 15%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure C-10. Mass balances for mesophilic CFCSTR acid-phase digester for meso-thermo CFCSTR two-phase Run TP15M-T conducted with Hanover Park sludge at a 15-day system HRT

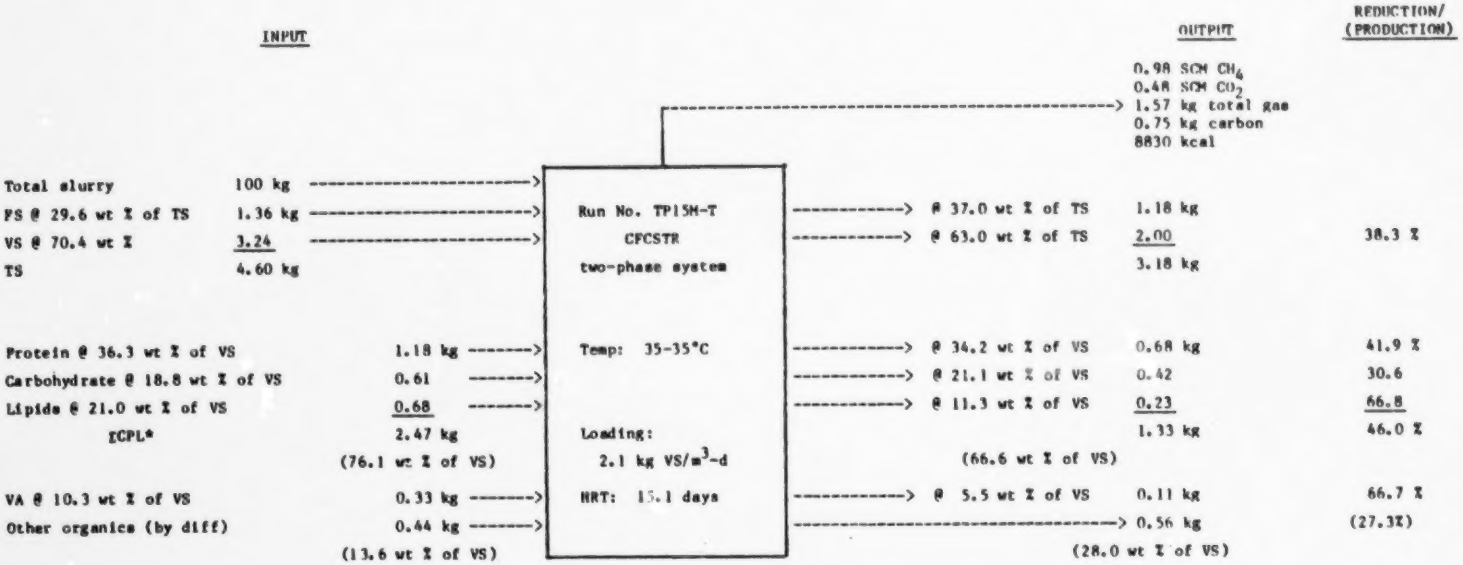


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.64 SCM CH_4
0.29 SCM CO_2
0.99 kg total gas
0.48 kg carbon
5790 kcal
Total slurry 100 kg
FS @ 31.2 wt % of TS 1.24 kg
VS @ 68.8 wt % 2.72
TS 3.96 kg
Run no. TP15M-T
CFCSTR
methane-phase
@ 37.0 wt % of TS 1.18 kg
@ 63.0 wt % of TS 2.00 26.5 %
3.18 kg
Protein @ 37.7 wt % of VS 1.03 kg
Carbohydrate @ 16.9 wt % of VS 0.46
Lipids @ 23.2 wt % of VS 0.63
ΣCPL* 2.12 kg
(77.8 wt % of VS)
Temp: 55°C
@ 34.2 wt % of VS 0.68 kg 33.4 %
@ 21.1 wt % of VS 0.42 8.1
@ 11.3 wt % of VS 0.23 64.1
1.33 kg 37.0 %
(66.6 wt % of VS)
Loading:
2.5 kg VS/m^3-d
HRT: 13.0 days
VA @ 4.2 wt % of VS 0.11 kg
Other organics (by diff) 0.49 kg
(18.0 wt % of VS)
@ 5.5 wt % of VS 0.11 kg 0.0 %
0.56 kg (14.3%)
(28.0 wt % of VS)

FS balance: effluent/feed = 95%
VS balance: effluent/feed = 110%
Carbon recovery in gas = 30%
Energy recovery in gas = 33%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure C-11. Mass balances for thermophilic CFCSTR methane-phase digester for meso-thermo CFCSTR two-phase Run TP15M-T conducted with Hanover Park sludge at a 15-day system HRT


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.98 SCM CH_4
0.48 SCM CO_2
1.57 kg total gas
0.75 kg carbon
8830 kcal
Total slurry 100 kg
FS @ 29.6 wt % of TS 1.36 kg
VS @ 70.4 wt % 3.24
TS 4.60 kg
Run No. TP15M-T
CFCSTR
two-phase system
@ 37.0 wt % of TS 1.18 kg
@ 63.0 wt % of TS 2.00 38.3 %
3.18 kg
Protein @ 36.3 wt % of VS 1.18 kg
Carbohydrate @ 18.8 wt % of VS 0.61
Lipids @ 21.0 wt % of VS 0.68
ΣCPL* 2.47 kg
(76.1 wt % of VS)
Temp: 35-35°C
@ 34.2 wt % of VS 0.68 kg 41.9 %
@ 21.1 wt % of VS 0.42 30.6
@ 11.3 wt % of VS 0.23 66.8
1.33 kg 46.0 %
(66.6 wt % of VS)
Loading:
2.1 kg VS/m^3-d
HRT: 15.1 days
VA @ 10.3 wt % of VS 0.33 kg
Other organics (by diff) 0.44 kg
(13.6 wt % of VS)
@ 5.5 wt % of VS 0.11 kg 66.7 %
0.56 kg (27.3%)
(28.0 wt % of VS)

FS balance: effluent/feed = 87%
VS balance: effluent/feed = 110%
Carbon recovery in gas = 40%
Energy recovery in gas = 42%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

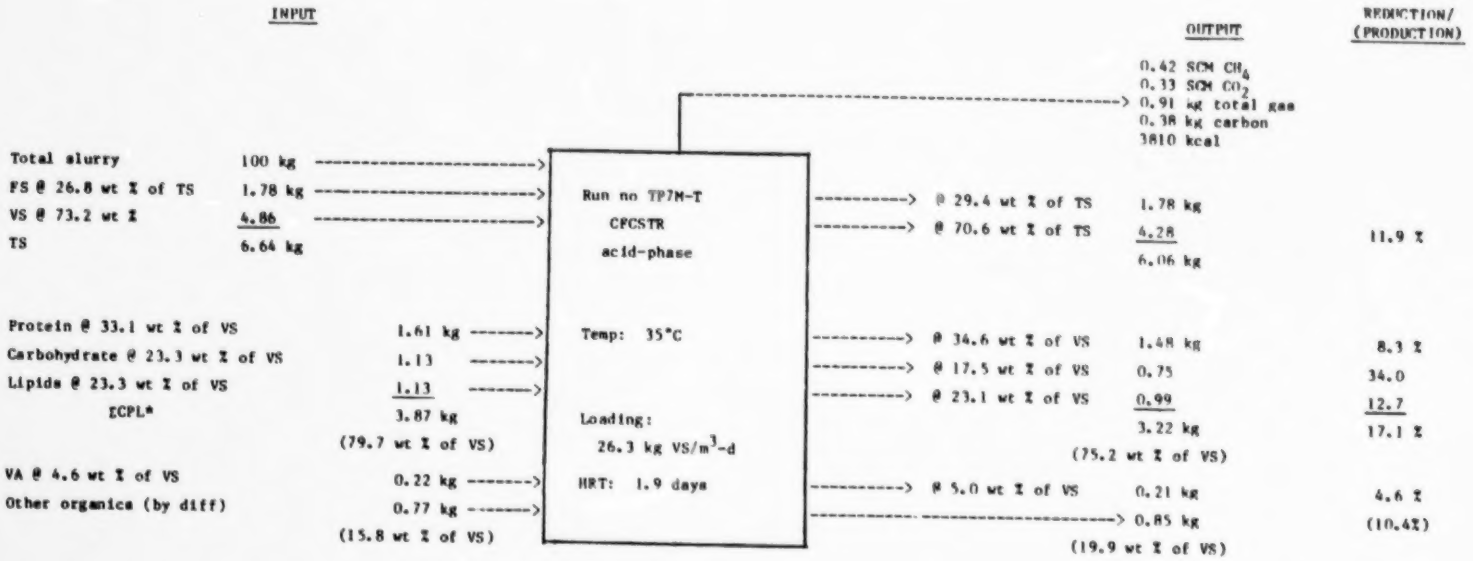
Figure C-12. Mass balances for meso-thermo CFCSTR two-phase Run TP15M-T conducted with Hanover Park sludge at a 15-day system HRT

TABLE C-9. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. TP7M-T: CFCSTR MESO-MESO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Digester No. 334)				
8/24/84	--	43.73	0.19	56.09
8/31/84	--	44.43	0.09	55.48
9/7/84	--	43.73	0.00	56.27
9/10/84	0.00	43.51	0.36	56.13
9/12/84	0.00	43.22	0.63	56.15
9/16/84	--	43.76	0.65	55.59
Methane-phase (Digester No. 331)				
8/24/84	--	31.22	0.09	68.69
8/31/84	--	30.96	0.61	68.43
9/7/84	--	32.51	0.03	67.45
9/10/84	--	32.98	0.49	66.53
9/12/84	0.00	32.55	0.16	67.29
9/16/84	--	33.38	0.26	66.36

TABLE C-10. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. TP7M-T: CFCSTR MESO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 7-DAY HRT

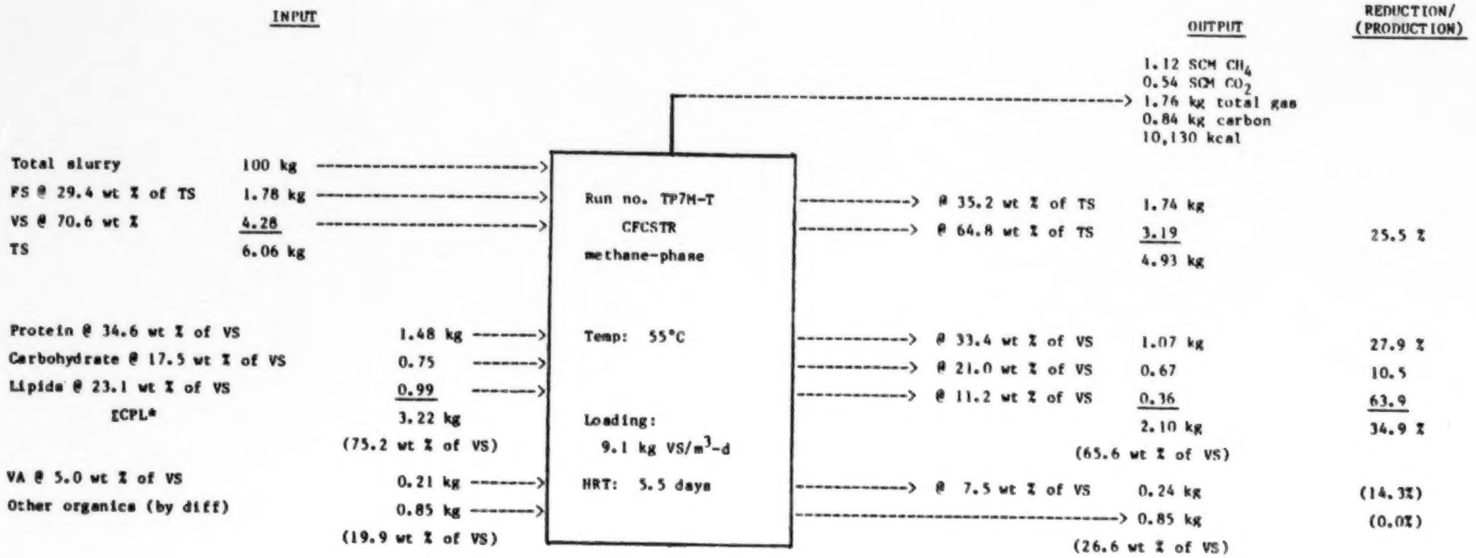
Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Digester No. 334)									
8/24/84	1,040	962	116	287	201	60	38	2,267	0
8/31/84	839	913	30	151	190	60	53	1,878	0
9/7/84	755	824	0	91	101	0	31	1,560	0
9/10/84	903	847	0	127	118	0	48	1,771	0
9/12/84	858	790	6	144	114	0	22	1,679	0
9/14/84	958	757	9	155	158	40	0	1,800	0
Methane-phase (Digester No. 331									
8/24/84	220	1,274	0	0	73	0	52	1,322	0
8/31/84	278	1,608	86	0	435	0	78	1,936	0
9/7/84	442	1,356	0	0	203	0	46	1,684	0
9/10/84	605	1,540	189	0	456	0	83	2,294	0
9/12/84	574	1,338	155	0	471	0	65	2,076	0
9/14/84	528	1,375	134	0	530	0	87	2,090	0


INPUT
OUTPUT
REDUCTION/
(PRODUCTION)
Total slurry
100 kg
FS @ 26.8 wt % of TS
1.78 kg
VS @ 73.2 wt %
4.86
TS
6.64 kg
Run no TP7M-T
CFCSTR
acid-phase
Temp: 35°C
Loading:
26.3 kg VS/m³-d
HRT: 1.9 days
0.42 SCM CH4
0.33 SCM CO2
0.91 kg total gas
0.38 kg carbon
3810 kcal
@ 29.4 wt % of TS
1.78 kg
@ 70.6 wt % of TS
4.28
11.9 %
6.06 kg
Protein @ 33.1 wt % of VS
1.61 kg
Carbohydrate @ 23.3 wt % of VS
1.13
Lipids @ 23.3 wt % of VS
1.13
ΣCPL*
3.87 kg
(79.7 wt % of VS)
@ 34.6 wt % of VS
1.48 kg
8.3 %
@ 17.5 wt % of VS
0.75
34.0
@ 23.1 wt % of VS
0.99
12.7
3.22 kg
17.1 %
(75.2 wt % of VS)
VA @ 4.6 wt % of VS
0.22 kg
Other organics (by diff)
0.77 kg
(15.8 wt % of VS)
@ 5.0 wt % of VS
0.21 kg
4.6 %
0.85 kg
(10.4%)
(19.9 wt % of VS)

FS balance: effluent/feed = 100%
VS balance: effluent/feed = 107%
Carbon recovery in gas = 13%
Energy recovery in gas = 12%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

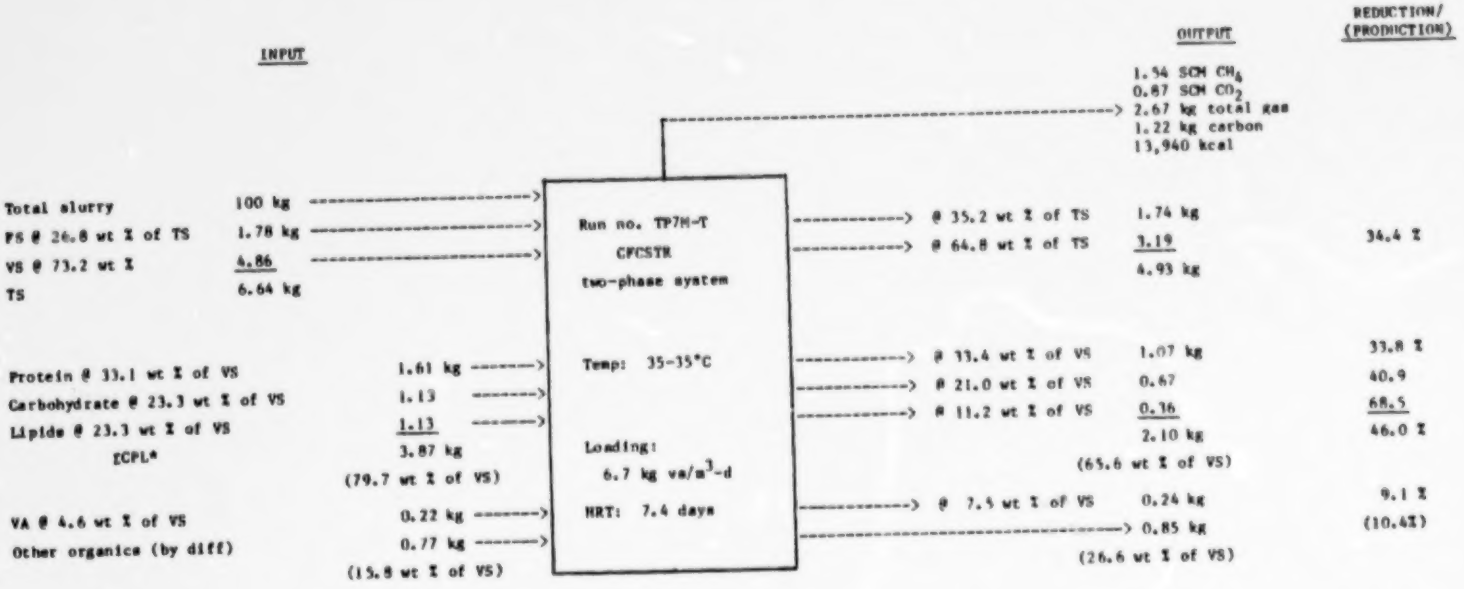
Figure C-13. Mass balances for mesophilic CFCSTR acid-phase digester for meso-thermo CFCSTR two-phase Run TP7M-T conducted with Hanover Park sluge at a 7-day system HRT


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
1.12 SCM CH4
0.54 SCM CO2
1.76 kg total gas
0.84 kg carbon
10,130 kcal
Total slurry 100 kg
FS @ 29.4 wt % of TS 1.78 kg
VS @ 70.6 wt % 4.28
TS 6.06 kg
Run no. TP7M-T
CFCSTR
methane-phase
@ 35.2 wt % of TS 1.74 kg
@ 64.8 wt % of TS 3.19 25.5 %
4.93 kg
Protein @ 34.6 wt % of VS 1.48 kg
Carbohydrate @ 17.5 wt % of VS 0.75
Lipids @ 23.1 wt % of VS 0.99
ΣCPL* 3.22 kg
(75.2 wt % of VS)
Temp: 55°C
Loading:
9.1 kg VS/m3-d
HRT: 5.5 days
@ 33.4 wt % of VS 1.07 kg 27.9 %
@ 21.0 wt % of VS 0.67 10.5
@ 11.2 wt % of VS 0.36 63.9
2.10 kg 34.9 %
(65.6 wt % of VS)
VA @ 5.0 wt % of VS 0.21 kg
Other organics (by diff) 0.85 kg
(19.9 wt % of VS)
@ 7.5 wt % of VS 0.24 kg (14.3%)
0.85 kg (0.0%)
(26.6 wt % of VS)

FS balance: effluent/feed = 98%
VS balance: effluent/feed = 116%
Carbon recovery in gas = 34%
Energy recovery in gas = 37%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure C-14. Mass balances for thermophilic CFCSTR methane-phase digester for meso-thermo CFCSTR two-phase Run TP7M-T conducted with Hanover Park sludge at a 7-day system HRT


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
1.54 SCM CH4
0.87 SCM CO2
2.67 kg total gas
1.22 kg carbon
13,940 kcal
Total slurry
100 kg
FS @ 26.8 wt % of TS
1.78 kg
VS @ 73.2 wt %
4.86
TS
6.64 kg
Run no. TP7M-T
CFCSTR
two-phase system
@ 35.2 wt % of TS
1.74 kg
@ 64.8 wt % of TS
3.19
34.4 %
4.93 kg
Protein @ 33.1 wt % of VS
1.61 kg
Carbohydrate @ 23.3 wt % of VS
1.13
Lipids @ 23.3 wt % of VS
1.13
ΣCPL*
3.87 kg
(79.7 wt % of VS)
Temp: 35-35°C
@ 33.4 wt % of VS
1.07 kg
33.8 %
@ 21.0 wt % of VS
0.67
40.9
@ 11.2 wt % of VS
0.36
68.5
2.10 kg
46.0 %
(65.6 wt % of VS)
Loading:
6.7 kg vs/m³-d
HRT: 7.4 days
VA @ 4.6 wt % of VS
0.22 kg
@ 7.5 wt % of VS
0.24 kg
9.1 %
Other organics (by diff)
0.77 kg
0.85 kg
(10.4%)
(15.8 wt % of VS)
(26.6 wt % of VS)

FS balance: effluent/feed = 98%
VS balance: effluent/feed = 121%
Carbon recovery in gas = 43%
Energy recovery in gas = 45%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure C-15. Mass balances for meso-thermo CFCSTR two-phase Run TP7M-T conducted with Hanover Park sludge at a 7-day system HRT

TABLE C-11. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. TP7T-T: CFCSTR THERMO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Digester No. 335)				
10/20/84	--	40.65	1.18	58.17
10/27/84	--	41.65	0.65	57.70
11/3/84	--	41.63	0.80	57.57
11/9/84	--	43.94	1.51	54.55
Methane-phase (Digester No. 331)				
10/20/84	--	28.40	0.19	71.41
10/27/84	--	28.45	0.68	70.87
11/3/84	--	30.01	0.31	69.68

TABLE C-12. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. TP7T-T: CFCSTR THERMO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Digester No. 335)									
10/19/84	718	594	167	134	355	14	0	1,622	0
10/26/84	1,229	865	260	248	572	0	94	2,660	0
11/2/84	1,036	710	219	211	468	0	0	2,180	0
Methane-phase (Digester No. 331)									
10/19/84	519	904	190	0	339	77	0	1,625	0
10/26/84	496	932	225	0	433	24	124	1,738	0
11/2/84	406	798	213	0	305	0	0	1,378	0

TABLE C-13. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. TP3T-T: CFCSTR THERMO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 3-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Digester No. 335)				
12/13/84	--	42.92	0.95	56.13
12/15/84	--	42.06	2.02	55.91
12/18/84	--	41.30	1.95	56.75
12/20/84	0.00	36.67	1.45	61.88
Methane-phase (Digester No. 331)				
12/13/84	--	30.06	0.00	69.94
12/15/84	--	32.23	0.59	67.18
12/18/84	--	32.29	0.00	67.71
12/20/84	0.00	30.92	0.00	69.08

TABLE C-14. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. TP3T-T: CFCSTR THERMO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 3-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Digester No. 334)									
12/13/84	1,272	761	230	302	468	176	0	2,630	0
12/15/84	1,393	704	218	307	506	208	105	2,796	0
12/18/84	1,475	853	256	400	519	134	0	2,997	0
12/20/84	627	348	82	64	289	139	87	1,306	0
Methane-phase (Digester No. 331)									
12/13/84	140	1,017	77	0	370	561	0	1,564	0
12/15/84	94	799	10	0	269	220	115	1,095	0
12/18/84	145	1,073	71	0	112	87	0	1,180	0
12/20/84	30	752	0	0	7	62	121	743	18

TABLE C-15. VOLATILE SOLIDS AND ORGANIC COMPONENT CONCENTRATIONS AND REDUCTIONS OBSERVED DURING STEADY-STATE MESO-MESO AND MESO-THERMO CFCSTR TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE[a]

Run	Sample	Sample date(s)[b]	Lot	Batch	TS	VS		Crude protein		Carbohydrates		Lipids		Organic reductions, %		
					mg/L	mg/L	wt % of TS	mg/L	wt % of VS	mg/L	wt % of VS	mg/L	wt % of VS	Protein	Carbohydrate	Lipids
TP15M-M	Feed	11/10/83	5	4	--	--	--	--	--	--	--	6200	22.06			
		11/11-11/14/83	5	4	39,350	28,100	71.41	9100	32.38	8307	29.56	--	--			
		11/13/83	5	4	--	--	--	--	--	--	--	7450	26.51			
			Run means[c]		39,350	28,100	71.41		32.38		29.56		24.28			
			Feed means[d]						30.81		29.56		24.28			
			Final means[e]					8880	31.60	8307	29.56	6821	24.28			
	Acid-phase effluent	11/5-11/7/83	5	4	37,280	25,660	68.83	--	--	5200	20.27	--	--			
		11/10/83	5	4	--	--	--	--	--	--	--	3564	11.96			
		11/11-11/14/83	5	4	--	--	--	--	--	--	--	3234	12.74			
		11/11-11/14/83	5	4	36,820	25,380	68.93	6060	23.88	5542	21.86	--	--			
					37,050	25,520	68.88	6094	23.88	5373	21.06	3458	11.35	31.37	35.30	49.23
	Methane-phase effluent	11/5/83	5	4	25,150	15,050	59.84	--	--	4256	28.21	--	--			
		11/10/83	5	4	--	--	--	--	--	--	--	2050	9.31			
		11/11-11/14/83	5	4	35,560	22,020	61.92	4969	22.57	4249	19.30	--	--			
		11/13/83	5	4	--	--	--	--	--	--	--	2000	9.08			
					30,335	18,535	61.06	4183	22.57	4406	23.76	1705	19.20	31.36	18.07	50.69
													System	52.89	46.98	75.01
TP7M-M	Feed	9/25-9/29/84	17	1	66,540	48,860	73.43	15,312	31.34	11,178	22.88	9314	19.06			
		9/25-9/29/84	17	1	66,540	48,860	73.43	--	--	10,610	21.72	--	--			
			Run means		66,540	48,860	73.43		31.34		22.30		19.06			
			Feed means						32.05		22.68		21.86			
			Final means					15,489	31.70	10,989	22.49	9997	20.46			
	Acid-phase effluent	9/25-9/29/84	17	1	60,720	42,480	69.96	14,006	32.97	8191	19.28	7178	16.90			
		9/25-9/29/84	17	1	60,720	42,480	69.96	--	--	7386	17.19	--	--			
					60,720	42,480	69.96	14,006	32.97	7791	18.34	7178	16.90	9.57	29.10	28.20
	Methane-phase effluent	9/25-9/29/84	17	1	50,690	32,810	64.73	11,531	35.14	5440	16.58	3824	11.65			
		9/25-9/29/84	17	1	50,690	32,810	64.73	--	--	4424	13.48	--	--			
					50,690	32,810	64.73	11,531	35.14	4932	15.03	3824	11.65	17.67	36.63	47.73
													System	25.55	55.07	61.75
TP3M-M[f]	Feed	12/17-12/21/84	21	1	68,500	46,600	68.03	17,975	38.57	10,459	21.80	8026	17.22			
		12/17-12/21/84	21	1	68,500	46,600	68.03	--	--	11,452	24.58	--	--			
			Run means		68,500	46,600	68.03		38.57		23.19		17.22			
			Feed means						38.57		23.19		17.22			
			Final means					17,975	38.57	10,955	23.51	8026	17.22			
	Acid-phase effluent	12/17-12/21/84	21	1	61,600	39,320	63.83	12,044	30.63	9399	23.90	6448	16.40			
		12/17-12/21/84	21	1	61,600	39,320	63.83	--	--	9056	23.03	--	--			
					61,600	39,320	63.83	12,044	30.63	9224	23.46	6448	16.40	33.00	15.80	19.66
	Methane-phase effluent	12/17-12/21/84	21	1	57,450	35,040	60.99	10,512	30.00	6206	17.71	5879	16.78			
		12/17-12/21/84	21	1	57,450	35,040	60.99	--	--	4622	13.19	--	--			
					57,450	35,040	60.99	10,512	30.00	5414	15.45	5879	16.78	12.72	41.32	8.82
													System	41.52	50.59	26.75

(continued)

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TABLE C-15 (continued)

Run	Sample	Sample date(s)[b]	Lot	Batch	TS	VS		Crude protein		Carbohydrates		Lipids		Organic reductions, %		
					mg/L	mg/L	wt % of TS	mg/L	wt % of VS	mg/L	wt % of VS	mg/L	wt % of VS	Protein	Carbohydrate	Lipids
TP15M-T	Feed	8/8-8/17/84	16	1	46,045	32,420	70.41	11,950	36.86	5943	18.33	7156	22.07			
			Run means		46,045	32,420	70.41		36.86		18.33		22.07			
			Feed means						35.71		19.27		20.00			
			Final means					11,762	36.28	6095	18.80	6821	21.04			
	Acid-phase effluent	8/8-8/17/84	16	1	39,590	27,240	68.81	10,262	37.67	4601	16.89	6250	22.94			
		8/8-8/17/84	16	1	39,590	27,240	68.81	--	--	--	--	6363	23.36			
					39,590	27,240	68.81	10,262	37.67	4601	16.89	6306	23.15	12.75	24.51	7.55
	Methane-phase effluent	8/8-8/17/84	16	1	31,790	20,020	62.98	6838	34.16	4228	21.12	2264	11.31			
					31,790	20,020	62.98	6838	34.16	4228	21.12	2264	11.31	33.37	8.11	64.10
													System	41.86	30.63	66.81
TP7M-T	Feed	9/10-9/14/84	17	1	66,430	48,640	73.22	16,638	34.21	12,202	25.09	11,994	24.66			
		9/10-9/14/84	17	1	66,430	48,640	73.22	--	--	11,114	22.85	--	--			
			Run means		66,430	48,640	73.22		34.21		23.97		24.66			
			Feed means						32,05		22.68		21.86			
			Final means					16,114	33.13	11,343	23.32	11,314	23.26			
	Acid-phase effluent	9/17/84	17	1	60,350	42,760	70.62	14,781	34.57	7918	18.52	9875	23.09			
		9/17/84	17	1	60,550	42,760	70.62	--	--	7091	16.50	--	--			
					60,550	42,760	70.62	14,781	34.57	7487	17.51	9875	23.09	8.27	33.99	12.72
	Methane-phase effluent	9/10-9/14/84	17	1	49,330	31,950	64.77	10,662	33.37	6880	21.53	3568	11.17			
		9/10-9/14/84	17	1	49,330	31,950	64.77	--	--	6520	20.41	--	--			
					49,330	31,950	64.77	10,662	33.37	6700	20.97	3568	11.17	27.87	10.51	63.87
													System	33.83	40.93	68.46

---

[a] Data reported are the averages of duplicate or triplicate determinations.

[b] A single sample date indicates that the analyses were conducted on a grab sample collected that day. A time period under this column indicates the start and end dates of collection of a grab or time-composite sample used for the analyses.

[c] Run means are the averages of the feed analyses conducted for a particular steady-state run on a single feed lot and batch.

[d] Feed means are the average organic contents (expressed as weight percent of VS) of all steady-state samples collected for a particular feed lot and batch.

[e] Final means are the average feed slurry organic concentrations and contents used to determine the organic reductions. The organic contents are the average of the feed and run organic contents. Organic feed concentrations were calculated as the product of the final mean organic feed contents and the average feed volatile solids concentration for the run.

[f] Steady-state run TP3M-M was conducted with mixed Downers Grove primary and Stickney activated sludges.

APPENDIX D

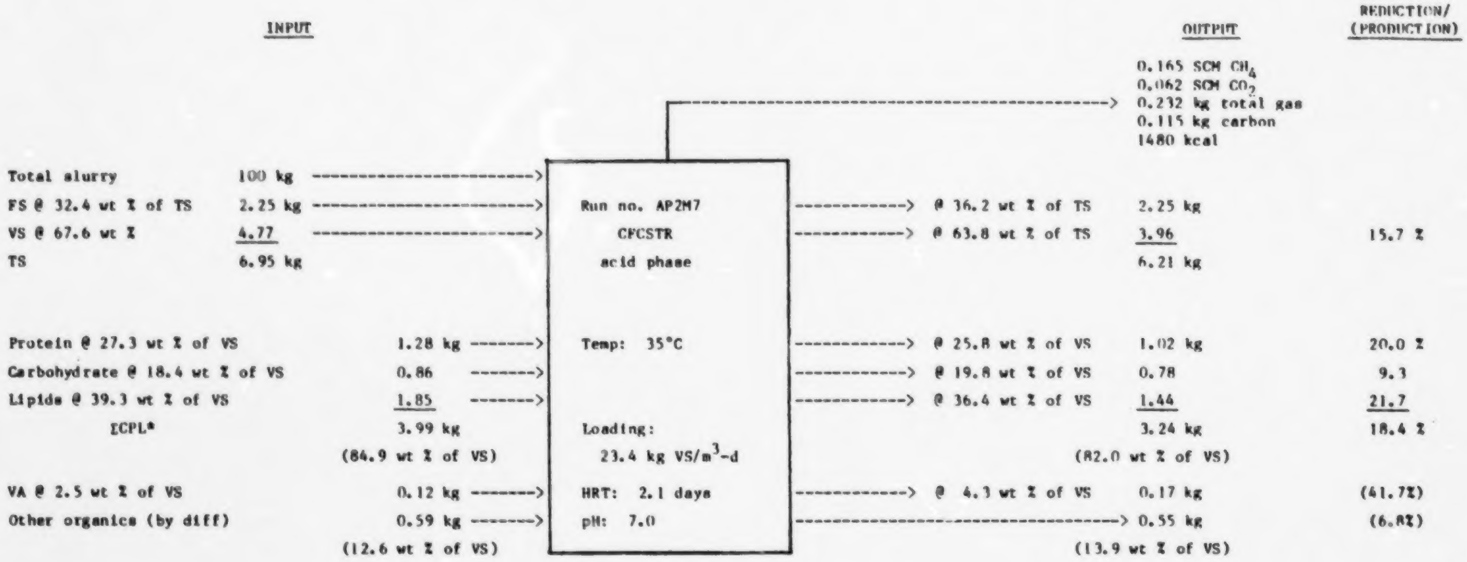
EFFLUENT ANALYSES FOR PARAMETRIC-EFFECT ACID-PHASE DIGESTERS

TABLE D-1. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP2M7: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 7 AND A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
12/4/83	--	31.45	2.12	66.43
12/11/83	--	24.77	1.13	74.10
12/19/83	--	33.62	1.40	64.98
1/2/84	--	35.52	1.11	63.37
1/10/84	--	22.41	1.28	76.31

TABLE D-2. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP2M7: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 7 AND A 2-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
12/5/83	529	577	81	115	167	0	0	1,228	0
12/12/83	610	706	97	133	166	0	0	1,437	0
12/19/83	528	693	102	133	182	0	6	1,360	0
1/3/84	817	539	107	99	213	22	0	1,532	0
1/10/84	826	716	114	108	247	42	4	1,730	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.165 SCM CH_4
0.062 SCM CO_2
0.232 kg total gas
0.115 kg carbon
1480 kcal
Total slurry 100 kg
FS @ 32.4 wt % of TS 2.25 kg
VS @ 67.6 wt % 4.77
TS 6.95 kg
Run no. AP2M7
CFCSTR
acid phase
@ 36.2 wt % of TS 2.25 kg
@ 63.8 wt % of TS 3.96 15.7 %
6.21 kg
Protein @ 27.3 wt % of VS 1.28 kg
Carbohydrate @ 18.4 wt % of VS 0.86
Lipids @ 39.3 wt % of VS 1.85
ΣCPL* 3.99 kg
(84.9 wt % of VS)
Temp: 35°C
Loading:
23.4 kg VS/m^3-d
HRT: 2.1 days
pH: 7.0
@ 25.8 wt % of VS 1.02 kg 20.0 %
@ 19.8 wt % of VS 0.78 9.3
@ 36.4 wt % of VS 1.44 21.7
3.24 kg 18.4 %
(82.0 wt % of VS)
VA @ 2.5 wt % of VS 0.12 kg
Other organics (by diff) 0.59 kg
(12.6 wt % of VS)
@ 4.3 wt % of VS 0.17 kg (41.7%)
0.55 kg (6.8%)
(13.9 wt % of VS)

FS balance: effluent/feed = 100%
VS balance: effluent/feed = 89%
Carbon recovery in gas = 4%
Energy recovery in gas = 5%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure D-1. Mass balances for mesophilic CFCSTR acid-phase Run AP2M7 conducted with Hanover Park sludge at pH 7 and about a 2-day HRT

TABLE D-3. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP2M6: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 6 AND A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
5/13/84	--	46.03	0.24	53.73
5/21/84	--	47.05	0.24	52.72
5/27/84	--	46.95	0.23	52.82

TABLE D-4. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP2M6: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 6 AND A 2-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
5/13/84	1,755	1,205	161	467	262	84	8	3,369	0
5/21/84	698	1,178	113	253	272	108	11	2,133	0
5/27/84	981	957	109	285	277	135	14	2,274	0

TABLE D-5. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP2M5.5: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5.5 AND A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
6/3/84	--	48.70	0.35	50.95

TABLE D-6. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP2M5.5 CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5.5 AND A 2-DAY HRT

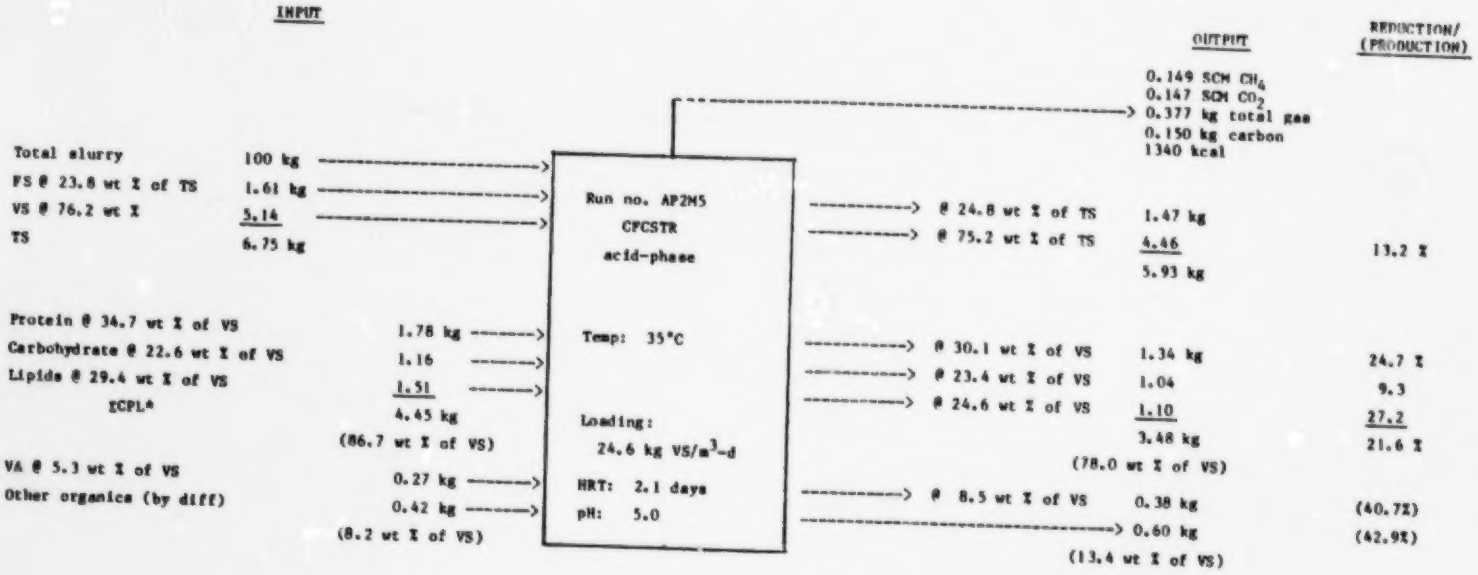
Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
6/3/84	1,465	1,223	160	547	1,002	185	39	3,657	0

TABLE D-7. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP2M5: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5 AND A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
6/10/84	--	46.91	0.71	52.38
6/12/84	--	50.01	0.60	49.39
6/14/84	--	49.82	0.94	49.25
6/16/84	0.00	49.82	0.81	49.36
6/19/84	0.00	48.69	0.70	50.60
6/22/84	--	51.12	0.69	48.19

TABLE D-8. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP2M5: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5 AND A 2-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
6/10/84	1,315	906	179	477	1,329	200	12	3,401	41
6/12/84	956	853	125	391	939	272	8	2,715	49
6/14/84	1,115	700	183	552	1,300	289	11	3,123	90
6/16/84	1,088	639	198	569	1,199	301	0	3,010	114
6/19/84	1,082	661	188	530	380	289	0	2,500	77
6/21/84	964	679	183	627	446	341	0	2,529	101


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
Total slurry 100 kg
FS @ 23.8 wt % of TS 1.61 kg
VS @ 76.2 wt % 5.14
TS 6.75 kg
Protein @ 34.7 wt % of VS 1.78 kg
Carbohydrate @ 22.6 wt % of VS 1.16
Lipids @ 29.4 wt % of VS 1.51
ΣCPL* 4.45 kg
(86.7 wt % of VS)
VA @ 5.3 wt % of VS 0.27 kg
Other organics (by diff) 0.42 kg
(8.2 wt % of VS)
Run no. AP2M5
CFCSTR
acid-phase
Temp: 35°C
Loading:
24.6 kg VS/m^3-d
HRT: 2.1 days
pH: 5.0
0.149 SCM CH$_4$
0.147 SCM CO$_2$
0.377 kg total gas
0.150 kg carbon
1340 kcal
@ 24.8 wt % of TS 1.47 kg
@ 75.2 wt % of TS 4.46
5.93 kg
13.2 %
@ 30.1 wt % of VS 1.34 kg 24.7 %
@ 23.4 wt % of VS 1.04 9.3
@ 24.6 wt % of VS 1.10 27.2
3.48 kg 21.6 %
(78.0 wt % of VS)
@ 8.5 wt % of VS 0.38 kg (40.7%)
0.60 kg (42.9%)
(13.4 wt % of VS)

FS balance: effluent/feed = 91%
VS balance: effluent/feed = 94%
Carbon recovery in gas = 5%
Energy recovery in gas = 4%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

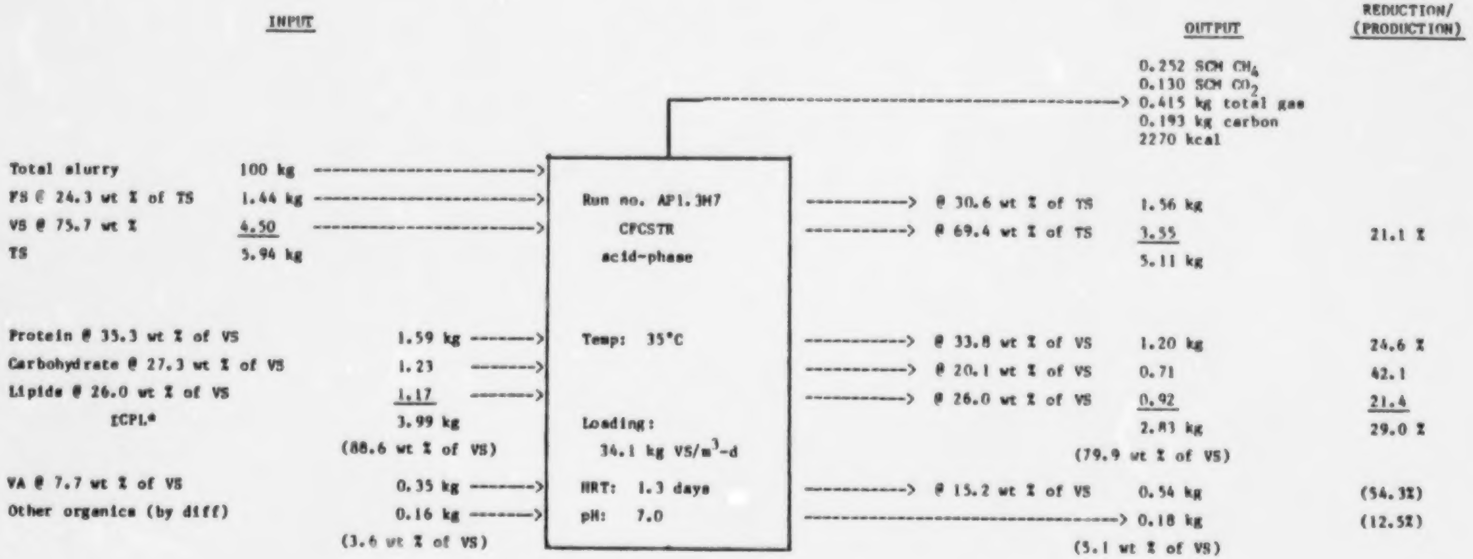
Figure D-2. Mass balances for mesophilic CFCSTR acid-phase Run AP2M5 conducted with Hanover Park sludge at pH 5 and about a 2-day HRT

TABLE D-9. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP1.3M7: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 7 AND A 1.3-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
3/25/84	--	34.37	0.88	64.76
4/4/84	--	32.66	0.63	66.71
4/8/84	--	28.58	0.59	70.83

TABLE D-10. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP1.3M7: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 7 AND A 1.3-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
3/30/84	2,549	1,556	237	477	379	91	0	4,573	0
3/31/84	2,454	1,604	261	572	380	97	0	4,603	0
4/4/84	2,491	1,764	277	559	386	85	0	4,767	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.252 SCM CH_4
0.130 SCM CO_2
0.415 kg total gas
0.193 kg carbon
2270 kcal
Total slurry 100 kg
FS @ 24.3 wt % of TS 1.44 kg
VS @ 75.7 wt % 4.50
TS 5.94 kg
Run no. AP1.3M7
CFCSTR
acid-phase
@ 30.6 wt % of TS 1.56 kg
@ 69.4 wt % of TS 3.55 21.1 %
5.11 kg
Protein @ 33.3 wt % of VS 1.59 kg
Carbohydrate @ 27.3 wt % of VS 1.23
Lipids @ 26.0 wt % of VS 1.17
ΣCPL* 3.99 kg
(88.6 wt % of VS)
Temp: 35°C
Loading:
34.1 kg VS/m^3-d
@ 33.8 wt % of VS 1.20 kg 24.6 %
@ 20.1 wt % of VS 0.71 42.1
@ 26.0 wt % of VS 0.92 21.4
2.83 kg 29.0 %
(79.9 wt % of VS)
VA @ 7.7 wt % of VS 0.35 kg
Other organics (by diff) 0.16 kg
(3.6 wt % of VS)
HRT: 1.3 days
pH: 7.0
@ 15.2 wt % of VS 0.54 kg (54.3%)
0.18 kg (12.5%)
(5.1 wt % of VS)

FS balance: effluent/feed = 108%

VS balance: effluent/feed = 88%

Carbon recovery in gas = 7%

Energy recovery in gas = 8%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

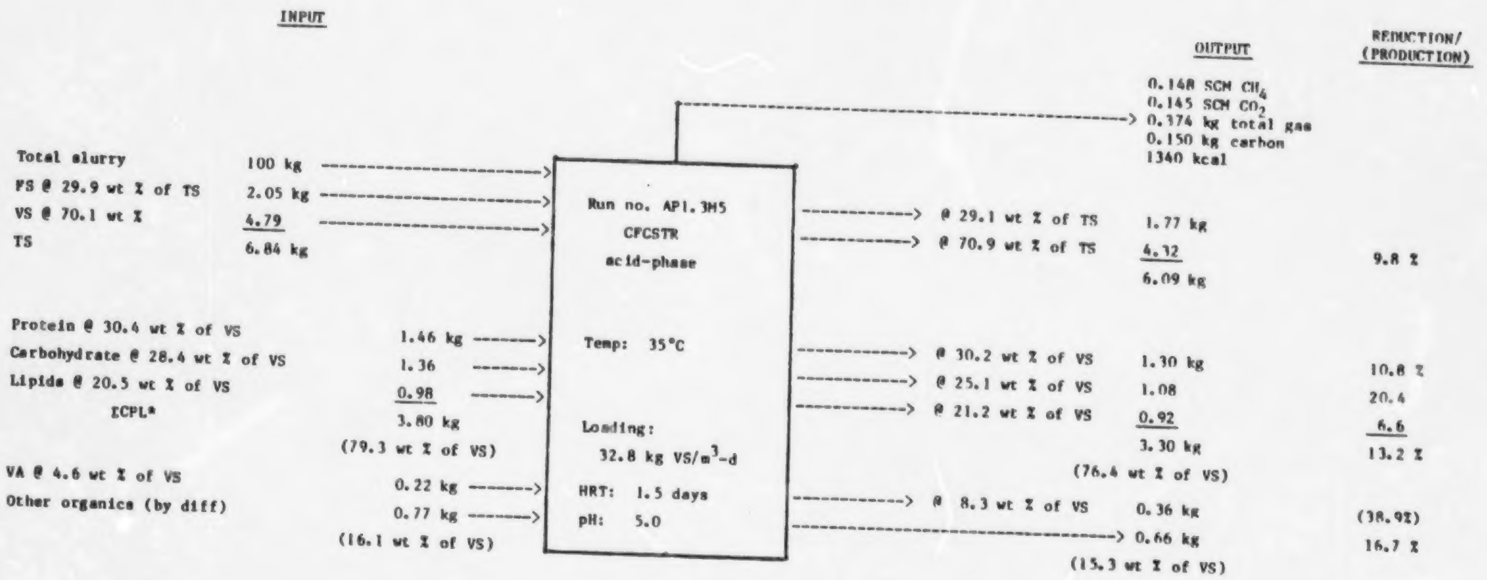
Figure D-3. Mass balances for mesophilic CFCSTR acid-phase Run AP1.3M7 conducted with Hanover Park sludge at pH 7 and about a 1.3-day HRT

TABLE D-11. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP1.3M5: CFCSTR MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5 AND A 1.3-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
6/24/84	--	51.77	0.70	47.53
7/1/84	0.00	49.71	0.56	49.73
7/2/84	0.00	47.47	1.10	51.43
7/3/84	0.11	46.45	0.84	52.60

TABLE D-12. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP1.3M5: CFCSTR THERMOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5 AND A 1.3-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
6/24/84	1,286	809	207	748	597	360	0	3,155	88
7/1/84	1,139	1,131	180	581	542	403	56	3,158	0
7/2/84	1,078	985	102	457	431	294	0	2,684	0
7/3/84	1,056	927	103	472	369	211	33	2,557	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
Total slurry 100 kg
FS @ 29.9 wt % of TS 2.05 kg
VS @ 70.1 wt % 4.79
TS 6.84 kg
Protein @ 30.4 wt % of VS 1.46 kg
Carbohydrate @ 28.4 wt % of VS 1.36
Lipids @ 20.5 wt % of VS 0.98
ΣCPL* 3.80 kg
(79.3 wt % of VS)
VA @ 4.6 wt % of VS 0.22 kg
Other organics (by diff) 0.77 kg
(16.1 wt % of VS)
Run no. AP1.3M5
CFCSTR
acid-phase
Temp: 35°C
Loading:
32.8 kg VS/m³-d
HRT: 1.5 days
pH: 5.0
0.148 SCM CH4
0.145 SCM CO2
0.374 kg total gas
0.150 kg carbon
1340 kcal
@ 29.1 wt % of TS 1.77 kg
@ 70.9 wt % of TS 4.32
6.09 kg 9.8 %
@ 30.2 wt % of VS 1.30 kg 10.8 %
@ 25.1 wt % of VS 1.08 20.4
@ 21.2 wt % of VS 0.92 6.6
3.30 kg 13.2 %
(76.4 wt % of VS)
@ 8.3 wt % of VS 0.36 kg (38.9%)
0.66 kg 16.7 %
(15.3 wt % of VS)

FS balance: effluent/feed = 86%
VS balance: effluent/feed = 98%
Carbon recovery in gas = 5%
Energy recovery in gas = 4%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

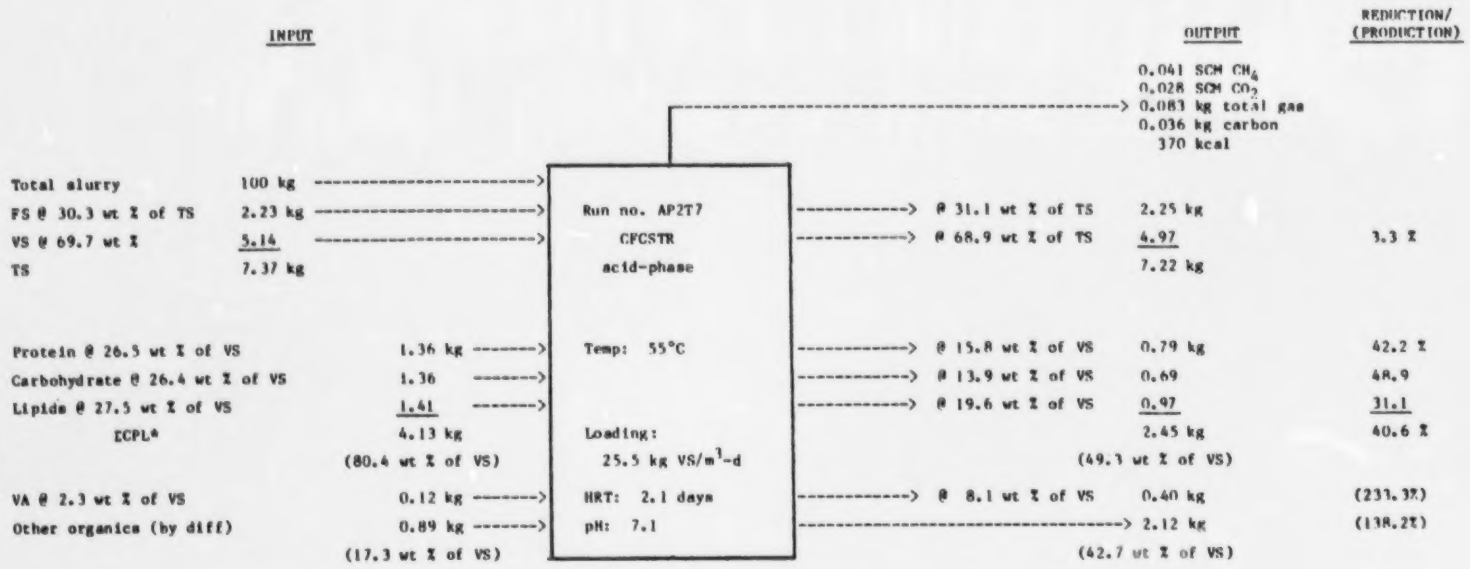
Figure D-4. Mass balances for mesophilic CFCSTR acid-phase Run AP1.3M5 conducted with Hanover Park sludge at pH 5 and about a 1.3-day HRT

TABLE D-13. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP2T7: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 7 AND A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
9/20/83	--	38.45	4.16	57.39
9/25/83	--	33.91	4.93	61.16
10/2/83	--	38.77	2.57	58.65
10/6/83	--	41.49	1.79	56.72
10/9/83	--	40.15	2.76	57.09
10/16/83	--	40.08	3.22	56.70
10/22/83	--	35.60	2.68	61.72
11/13/83	--	34.11	1.30	64.59
11/20/83	--	37.84	2.88	59.27

TABLE D-14. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP2T7: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE ONDUCTED AT pH 7 AND A 2-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
9/18/83	1,531	1,011	342	565	804	109	0	3,505	
9/26/83	1,443	857	272	403	578	33	0	2,957	
10/3/83	1,458	875	307	391	643	33	0	3,041	
10/17/83	1,372	989	306	378	625	35	0	3,028	
10/21/83	1,379	924	324	438	676	30	0	3,062	
10/29/83	1,480	1,109	427	535	645	29	0	3,430	
11/7/83	1,462	1,094	371	475	739	42	0	3,384	
11/15/83	1,434	1,031	337	499	780	86	0	3,349	


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.041 SCM CH_4
0.028 SCM CO_2
0.083 kg total gas
0.036 kg carbon
370 kcal
Total slurry 100 kg
FS @ 30.3 wt % of TS 2.23 kg
VS @ 69.7 wt % 5.14
TS 7.37 kg
Run no. AP2T7
CFCSTR
acid-phase
@ 31.1 wt % of TS 2.25 kg
@ 68.9 wt % of TS 4.97 3.3 %
7.22 kg
Protein @ 26.5 wt % of VS 1.36 kg
Carbohydrate @ 26.4 wt % of VS 1.36
Lipids @ 27.5 wt % of VS 1.41
ΣCPL* 4.13 kg
(80.4 wt % of VS)
Temp: 55°C
Loading:
25.5 kg VS/m^3-d
@ 15.8 wt % of VS 0.79 kg 42.2 %
@ 13.9 wt % of VS 0.69 48.9
@ 19.6 wt % of VS 0.97 31.1
2.45 kg 40.6 %
(49.3 wt % of VS)
VA @ 2.3 wt % of VS 0.12 kg
Other organics (by diff) 0.89 kg
(17.3 wt % of VS)
HRT: 2.1 days
pH: 7.1
@ 8.1 wt % of VS 0.40 kg (233.3%)
2.12 kg (138.2%)
(42.7 wt % of VS)

FS balance: effluent/feed = 101%
VS balance: effluent/feed = 98%
Carbon recovery in gas = 1%
Energy recovery in gas = 1%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure D-5. Mass balances for thermophilic CFCSTR acid-phase Run AP2T7 conducted with Hanover Park sludge at pH 7 and about a 2-day HRT

TABLE D-15. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP2T6: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 6 AND A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
5/27/84	--	51.69	1.05	47.26

TABLE D-16. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP2T6: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 6 AND A 2-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
5/27/84	2,450	1,167	407	588	818	73	24	4,610	0

TABLE D-17. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP2T5.5: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5.5 AND A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
5/6/84	--	65.85	6.80	27.35
5/13/84	--	54.00	1.25	44.76

TABLE D-18. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP2T5.5: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5.5 AND A 2-DAY HRT

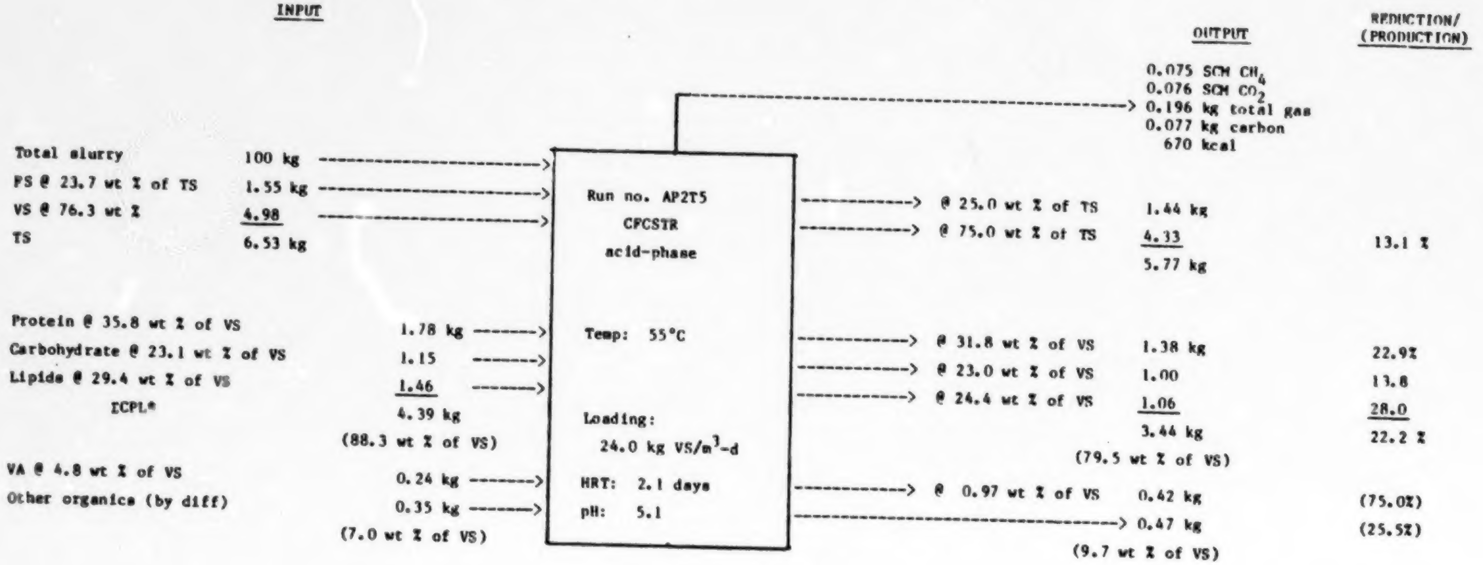
Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
5/7/84	1,639	1,019	173	542	365	105	17	3,238	0
5/13/84	2,249	1,438	266	1,124	459	182	21	4,750	47

TABLE D-19. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP2T5: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5 AND A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
6/10/84	--	48.67	1.19	50.15
6/12/84	--	49.59	1.50	48.92
6/14/84	--	49.64	1.88	48.48
6/16/84	0.16	48.41	1.73	49.70
6/19/84	0.22	49.88	1.41	48.50

TABLE D-20. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP2T5: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5 AND A 2-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
6/10/84	1,789	727	154	341	920	4	3	3,260	55
6/12/84	1,952	873	182	393	1,041	66	0	3,702	73
6/16/84	2,107	884	182	375	748	54	0	3,674	128
6/19/84	2,054	895	190	393	227	49	0	3,340	88


INPUT
OUTPUT
REDUCTION/
(PRODUCTION)
0.075 SCM CH$_4$
0.076 SCM CO$_2$
0.196 kg total gas
0.077 kg carbon
670 kcal
Total slurry 100 kg
FS @ 23.7 wt % of TS 1.55 kg
VS @ 76.3 wt % 4.98
TS 6.53 kg
Run no. AP2T5
CFCSTR
acid-phase
@ 25.0 wt % of TS 1.44 kg
@ 75.0 wt % of TS 4.33 13.1 %
5.77 kg
Protein @ 35.8 wt % of VS 1.78 kg
Carbohydrate @ 23.1 wt % of VS 1.15
Lipids @ 29.4 wt % of VS 1.46
ΣCPL* 4.39 kg
(88.3 wt % of VS)
Temp: 55°C
@ 31.8 wt % of VS 1.38 kg 22.9%
@ 23.0 wt % of VS 1.00 13.8
@ 24.4 wt % of VS 1.06 28.0
3.44 kg 22.2 %
(79.5 wt % of VS)
Loading:
24.0 kg VS/m^3-d
HRT: 2.1 days
pH: 5.1
VA @ 4.8 wt % of VS 0.24 kg
Other organics (by diff) 0.35 kg
(7.0 wt % of VS)
@ 0.97 wt % of VS 0.42 kg (75.0%)
0.47 kg (25.5%)
(9.7 wt % of VS)

FS balance: effluent/feed = 93%
VS balance: effluent/feed = 91%
Carbon recovery in gas = 3%
Energy recovery in gas = 2%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

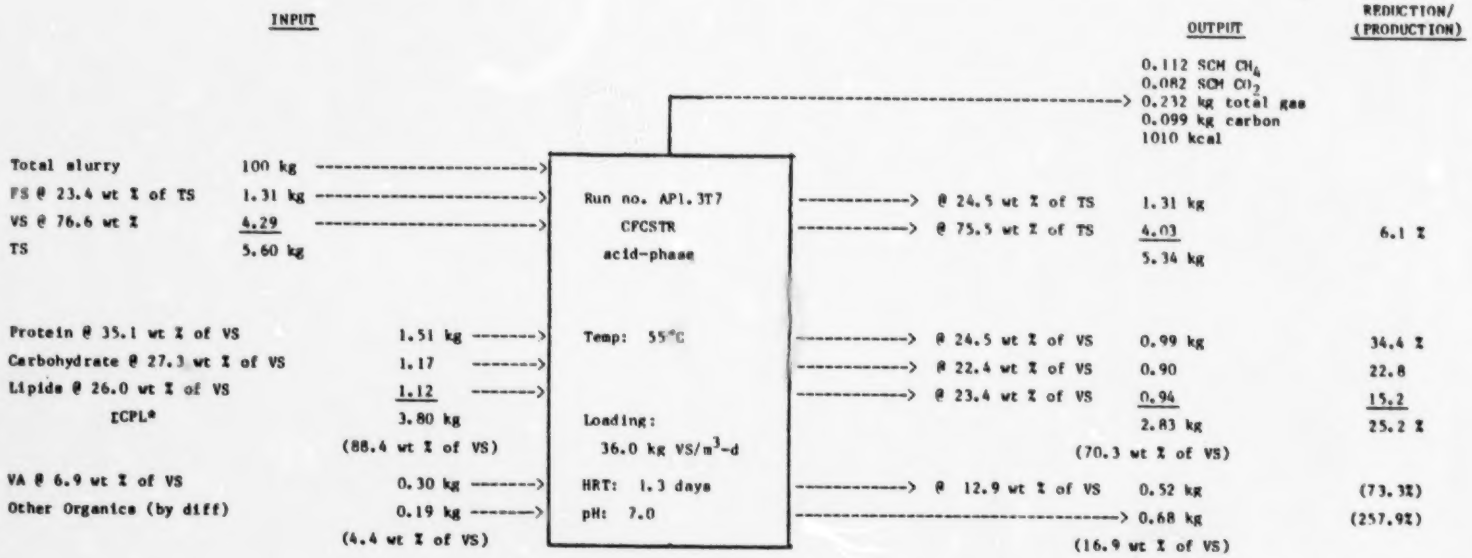
Figure D-6. Mass balances for thermophilic CFCSTR acid-phase Run AP2T5 conducted with Hanover Park sludge at pH 5 and about a 2-day HRT

TABLE D-21. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP1.3T7: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 7 AND A 1.3-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
2/21/84	--	37.51	1.79	60.70
3/5/84	--	40.20	0.92	58.88
3/12/84	--	43.05	0.73	56.22
3/25/84	--	39.98	0.51	59.52

TABLE D-22. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP1.3T7: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 7 AND A 1.3-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
2/20/84	1,773	997	373	652	753	52	0	3,753	0
2/27/84	1,722	1,109	400	653	1,064	0	0	3,964	0
3/5/84	1,813	1,127	400	659	1,051	0	0	4,066	0
3/12/84	1,851	1,738	446	819	1,117	175	0	4,881	0
4/29/84	1,620	1,559	459	662	933	104	0	4,257	0


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
0.112 SCM CH4
0.082 SCM CO2
0.232 kg total gas
0.099 kg carbon
1010 kcal
Total slurry 100 kg
FS @ 23.4 wt % of TS 1.31 kg
VS @ 76.6 wt % 4.29
TS 5.60 kg
Run no. AP1.3T7
CFCSTR
acid-phase
@ 24.5 wt % of TS 1.31 kg
@ 75.5 wt % of TS 4.03 6.1 %
5.34 kg
Protein @ 35.1 wt % of VS 1.51 kg
Carbohydrate @ 27.3 wt % of VS 1.17
Lipids @ 26.0 wt % of VS 1.12
ΣCPL* 3.80 kg
(88.4 wt % of VS)
Temp: 55°C
Loading:
36.0 kg VS/m³-d
HRT: 1.3 days
pH: 7.0
@ 24.5 wt % of VS 0.99 kg 34.4 %
@ 22.4 wt % of VS 0.90 22.8
@ 23.4 wt % of VS 0.94 15.2
2.83 kg 25.2 %
(70.3 wt % of VS)
VA @ 6.9 wt % of VS 0.30 kg
Other Organics (by diff) 0.19 kg
(4.4 wt % of VS)
@ 12.9 wt % of VS 0.52 kg (73.3%)
0.68 kg (257.9%)
(16.9 wt % of VS)

FS balance: effluent/feed = 100%
VS balance: effluent/feed = 99%
Carbon recovery in gas = 4%
Energy recovery in gas = 4%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

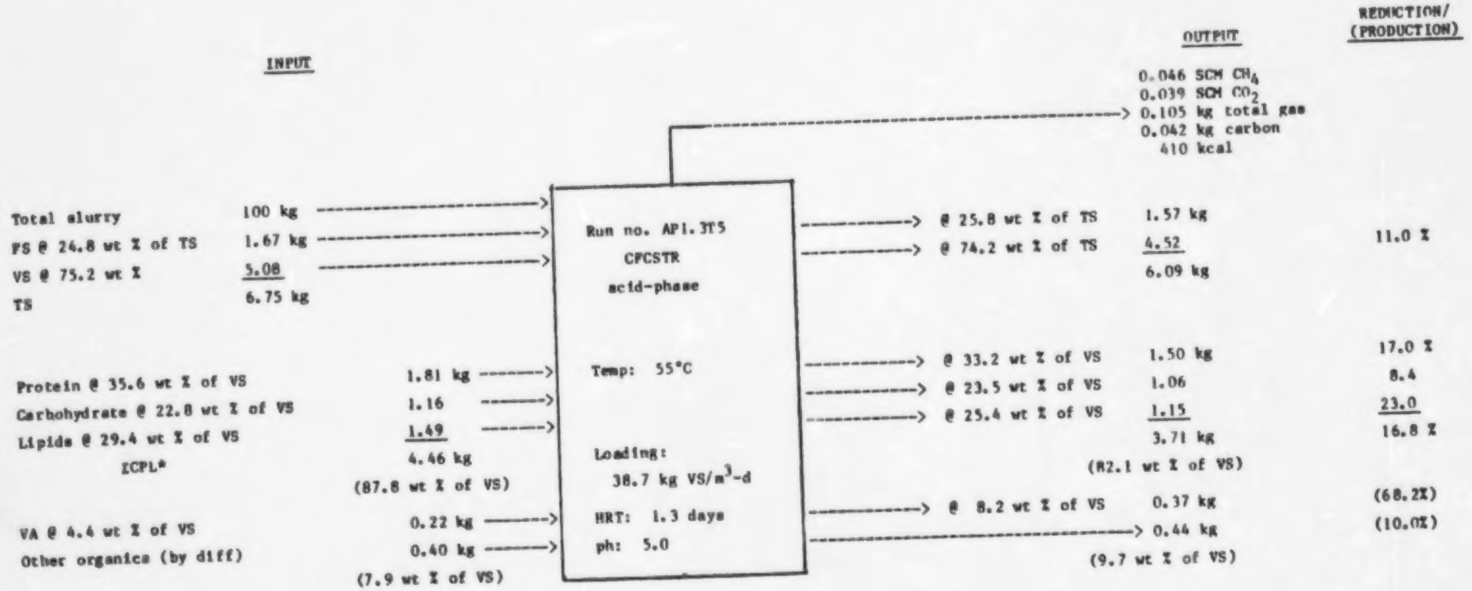
Figure D-7. Mass balances for thermophilic CFCSTR acid-phase Run AP1.3T7 conducted with Hanover Park sludge at pH 7 and about a 1.3-day HRT

TABLE D-23. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. AP1.3T5: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5 AND A 1.3-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
6/24/84	--	47.54	2.75	49.71
7/1/84	0.30	45.99	2.45	51.26
7/2/84	0.37	45.29	2.46	51.88
7/3/84	0.60	45.63	2.16	51.61

TABLE D-24. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. AP1.3T5: CFCSTR THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT pH 5 AND A 1.3-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
6/21/84	1,966	797	168	420	249	98	0	3,217	115
6/24/84	2,072	792	190	434	333	96	0	3,392	97
7/1/84	2,023	824	171	400	330	101	73	3,372	85
7/2/84	1,722	731	120	366	338	109	0	2,909	74


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
Total slurry 100 kg
FS @ 24.8 wt % of TS 1.67 kg
VS @ 75.2 wt % 5.08
TS 6.75 kg
Protein @ 35.6 wt % of VS 1.81 kg
Carbohydrate @ 22.8 wt % of VS 1.16
Lipids @ 29.4 wt % of VS 1.49
ΣCPL* 4.46 kg
(87.8 wt % of VS)
VA @ 4.4 wt % of VS 0.22 kg
Other organics (by diff) 0.40 kg
(7.9 wt % of VS)
Run no. AP1.3T5
CFCSTR
acid-phase
Temp: 55°C
Loading:
38.7 kg VS/m³-d
HRT: 1.3 days
ph: 5.0
0.046 SCM CH4
0.039 SCM CO2
0.105 kg total gas
0.042 kg carbon
410 kcal
@ 25.8 wt % of TS 1.57 kg
@ 74.2 wt % of TS 4.52 11.0 %
6.09 kg
@ 33.2 wt % of VS 1.50 kg 17.0 %
@ 23.5 wt % of VS 1.06 8.4
@ 25.4 wt % of VS 1.15 23.0
3.71 kg 16.8 %
(82.1 wt % of VS)
@ 8.2 wt % of VS 0.37 kg (68.2%)
0.44 kg (10.0%)
(9.7 wt % of VS)

FS balance: effluent/feed = 94%
VS balance: effluent/feed = 91%
Carbon recovery in gas = 1%
Energy recovery in gas = 1%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure D-8. Mass balances for thermophilic CFCSTR acid-phase Run AP1.3T5 conducted with Hanover Park sludge at pH 5 and about a 1.3-day HRT

TABLE D-25. VOLATILE SOLIDS AND ORGANIC COMPONENT CONCENTRATIONS AND REDUCTIONS OBSERVED DURING STEADY-STATE MESOPHILIC (35°C) AND THERMOPHILIC (55°C) CFCSTR ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE WITH pH CONTROL[a]

Run	Sample	Sample date(s)[b]	Lot	Batch	TS	VS		Crude protein		Carbohydrates		Lipids		Organic reductions, %		
					mg/L	mg/L	wt % of TS	mg/L	wt % of VS	mg/L	wt % of VS	mg/L	wt % of VS	Protein	Carbohydrate	Lipids
AP2M7	Feed	12/9-12/14/83	6	2	69,460	46,930	67.56	12,775	27.22	--	--	19,068	40.63			
			Run means[c]		69,460	46,930	67.56		27.22				40.63			
			Feed means[d]						27.30		18.44		37.98			
			Final means[e]					12,793	27.26	8654	18.44	18,443	39.30			
	Effluent	12/9-12/14/83	6	2	62,120	39,650	63.83	10,238	25.82	7992	20.16	14,444	36.43			
		12/9-12/14/83	6	2	62,120	39,650	63.83	--	--	7708	19.44	--	--			
					62,120	39,650	63.83	10,238	25.82	7851	19.80	14,444	36.43	19.97	9.28	21.68
AP2M5	Feed	6/19-6/23/84	13	1	67,530	51,460	76.20	17,625	34.25	10,994	21.36	15,104	29.35			
		6/19-6/23/84	13	1	67,350	51,460	76.20	--	--	11,813	22.96	--	--			
			Run means		67,530	51,460	76.20		34.25		22.16		29.35			
			Feed means						35.09		23.13		29.35			
			Final means					17,841	34.67	11,650	22.64	15,104	29.35			
	Effluent	6/19-6/23/84	13	1	59,290	44,590	76.35	13,438	30.14	10,561	23.37	10,988	24.64			
					59,290	44,590	76.35	13,438	30.14	10,561	23.37	10,988	24.64	24.68	9.35	27.25
AP1.3M7	Feed	3/28-4/6/84	8	5	59,380	44,960	75.72	16,019	35.63	12,724	28.30	11,736	26.10			
			Run means		59,380	44,960	75.72		35.63		28.30		26.10			
			Feed means						35.00		26.32		25.98			
			Final means					15,880	35.32	12,279	27.31	11,708	26.04			
	Effluent	3/28-4/6/84	8	5	51,060	35,410	69.35	11,975	33.82	6917	19.53	9202	25.99			
		3/38-4/6/84	8	5	51,060	35,410	69.35	--	--	7311	20.65	--	--			
					51,060	35,410	69.35	11,975	33.82	7114	20.09	9202	25.99	24.59	42.06	21.40
AP1.3M5	Feed	7/2-7/6/84	14	2	68,440	47,970	70.09	14,594	30.42	13,637	28.43	9824	20.48			
			Run means		68,440	47,970	70.09		30.42		28.43		20.48			
			Feed means						30.42		28.43		20.48			
			Final means					14,594	30.42	13,637	28.43	9824	20.48			
	Effluent	7/2-7/6/84	14	2	60,890	43,200	70.95	13,025	30.15	10,849	25.11	9176	21.24			
					60,890	43,200	70.95	13,025	30.15	10,849	25.11	9176	21.24	10.75	20.44	6.60
AP2T7	Feed	10/3/83	5	3	77,930	53,440	68.57	14,031	26.26	14,100	26.38	14,600	27.32			
		10/14/83	5	3	71,600	50,280	70.22	--	--	13,300	26.45	--	--			
		10/14/83	5	3	71,600	50,280	70.22	13,112	26.08	--	--	14,100	28.04			
			Run means		73,710	51,333	69.67		26.17		26.42		27.68			
			Feed means						26.91		26.48		27.36			
			Final means					13,624	26.54	13,578	26.45	14,127	27.52			
		10/3/83	5	3	73,730	50,050	67.88	7994	15.97	--	--	9700	19.38			
		10/14/83	5	3	70,760	49,640	69.87	7633	15.66	6932	13.94	9956	19.76			
					72,245	49,745	68.86	7870	15.82	6934	13.94	9735	19.57	42.23	48.93	31.09

(continued)

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TABLE D-25 (continued)

Run	Sample	Sample date(s)[b]	Lot	Batch	TS	VS		Crude protein		Carbohydrates		Lipids		Organic reductions, %		
					mg/L	mg/L	wt % of TS	mg/L	wt % of VS	mg/L	wt % of VS	mg/L	wt % of VS	Protein	Carbohydrate	Lipids
AP2T5	Feed	6/12-6/16/84	13	1	65,310	49,840	76.31	18,238	36.59	--	--	--	--			
			Run means		65,310	49,840	76.31		36.59		--		--			
			Feed means						35.09		23.13		29.35			
			Final means					17,863	35.84	11,528	23.13	14,628	29.35			
	Effluent	6/12-6/16/84	13	1	57,710	43,260	75.98	13,775	31.84	10,116	23.38	10,539	24.36			
		6/12-6/16/84	13	1	57,710	43,260	75.98	--	--	11,230	25.96	--	--			
		6/12-6/16/84	13	1	57,710	43,260	75.98	--	--	8479	19.60	--	--			
					57,710	43,260	75.98	13,775	31.84	9941	22.98	10,539	24.36	22.89	13.75	27.95
AP1.3T7	Feed	3/18-3/27/84	8	5	55,960	42,890	76.64	15,125	35.26	12,118	28.25	--	--			
			Run means		55,960	42,890	76.64		35.26		28.25		--			
			Feed means						35.00		26.32		25.98			
			Final means					15,067	35.13	11,700	27.28	11,143	25.98			
	Effluent	3/18-3/27/84	8	5	53,410	40,340	75.53	9885	24.50	8478	21.02	9451	23.43			
		3/18-3/27/84	8	5	53,410	40,340	75.53	--	--	9595	23.79	--	--			
					53,410	40,340	75.53	9885	24.50	9036	22.40	9451	23.43	34.39	22.72	15.18
AP1.3T5	Feed	6/28-7/2/84	13	1	67,530	50,750	75.15	18,331	36.12	10,786	21.25	--	--			
		6/28-7/2/84	13	1	67,530	50,750	75.15	--	--	12,016	23.68	--	--			
			Run means		67,530	50,750	75.15		36.12		24.28		--			
			Feed means						35.09		23.13		29.35			
			Final means					18,067	35.60	11,571	22.80	14,895	29.35			
	Effluent	6/28-7/2/84	13	1	60,860	45,170	74.22	14,988	33.18	10,595	23.46	11,466	25.38			
					60,860	45,170	74.22	14,988	33.18	10,595	23.46	11,466	25.38	17.04	8.43	23.02

[a] Date reported are the averages of duplicate or triplicate determinations.

[b] A single sample date indicates that the analyses were conducted on a grab sample collected that day. A time period under this column indicates the start and end dates of collection of a grab or time-composite sample used for the analyses.

[c] Run means are the averages of the feed analyses conducted for a particular steady-state run on a single feed lot and batch.

[d] Feed means are the average organic contents (expressed as weight percent of VS) of all steady-state samples collected for a particular feed lot and batch.

[e] Final means are the average feed slurry organic concentrations and contents used to determine the organic reductions. The organic contents are the average of the feed and run organic contents. Organic feed concentrations were calculated as the product of the final mean organic feed contents and the average feed volatile solids concentration for the run.

APPENDIX E

EFFLUENT ANALYSES FOR ADVANCED TWO-PHASE DIGESTION SYSTEMS

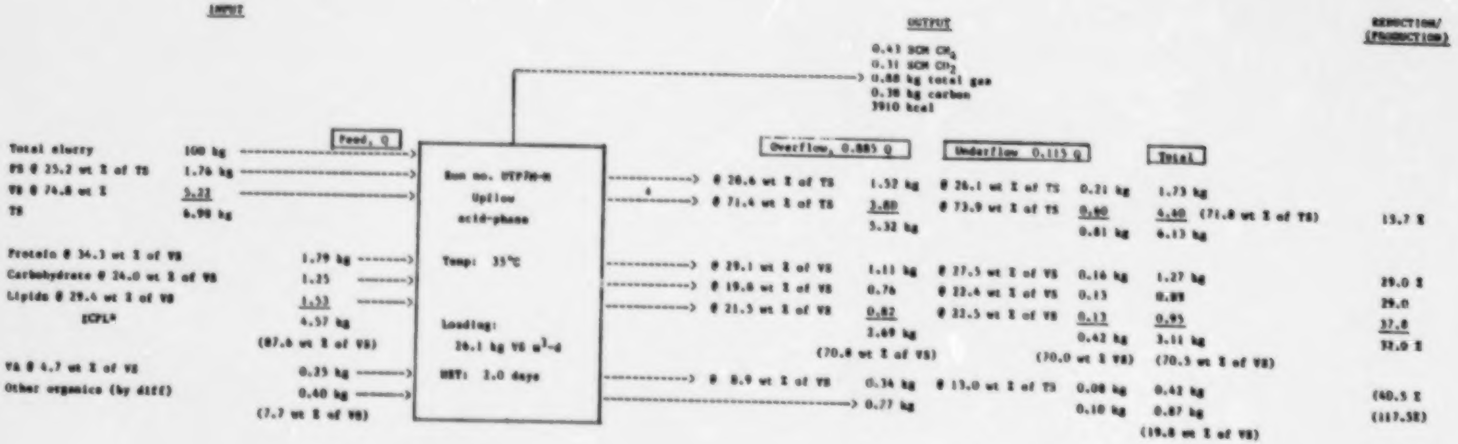
TABLE E-1. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. UTP7M-M: UPFLOW MESO-MESO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Digester No. 338)				
5/30/84	--	41.27	0.90	57.83
6/4/84	--	41.23	1.35	57.41
6/14/84	--	42.57	1.30	56.13
6/16/84	--	41.44	0.54	58.02
6/18/84	--	38.30	0.62	61.08
6/23/84	--	47.86	0.88	51.26
6/26/84	--	39.76	0.45	59.78
6/29/84	--	40.31	0.13	59.56
7/2/84	0.36	42.39	0.21	57.03
Methane-phase (Digester No. 339)				
5/30/84	--	32.14	1.41	66.45
6/4/84	--	31.77	0.91	67.32
6/14/84	--	31.83	0.89	67.28
6/16/84	--	32.87	0.05	67.08
6/18/84	--	32.15	0.45	67.40
6/23/84	--	32.13	0.22	67.65
6/26/84	--	31.65	0.23	68.12
6/29/84	--	32.65	0.00	67.35
7/2/84	0.00	32.11	0.11	67.78

TABLE E-2. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. UTP7M-M: UPFLOW MESO-MESO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Sample location	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Digester No. 338)										
6/28/84	Left Chamber*	2589	1206	390	1061	457	248	43	4993	79
6/30/84	Left chamber	2564	1358	239	919	278	187	35	4746	99
6/14/84	Underflow	2523	1406	420	1204	649	440	49	5435	0
6/17/84	Underflow	2325	1536	440	947	772	370	51	5214	35
6/24/84	Underflow	2713	1565	498	1373	908	375	0	6011	0
6/28/84	Underflow	1832	1667	303	602	422	235	34	4204	0
6/30/84	Underflow	3175	1607	563	1496	855	424	42	6655	70
7/3/84	Underflow	2714	1278	452	1085	789	490	40	5570	24
5/3/84	Overflow	1163	925	242	335	1032	119	64	3016	0
6/4/84	Overflow	945	710	144	187	686	72	0	2192	0
6/14/84	Overflow	1559	1199	330	604	465	258	34	3610	0
6/17/84	Overflow	1659	1232	314	580	513	233	39	3726	28
6/24/84	Overflow	1085	1682	307	350	580	207	0	3359	0
6/28/84-7/1/84	Overflow	1224	1249	268	222	310	191	82	2908	0
7/3/84	Overflow	1302	1033	254	360	478	265	42	3016	0
Methane-phase (Digester No. 339)										
6/28/84	Bottom port	0	0	0	0	0	0	0	0	0
6/30/84	Bottom port	6	0	0	0	0	0	0	6	0
6/28/84	11.5-L port	168	436	0	0	0	6	8	529	0
6/30/84	11.5-L port	178	144	0	0	0	0	0	295	0
6/28/84	15.5-L port	198	448	0	0	0	6	0	564	0
6/30/84	15.5-L port	224	174	0	0	0	0	8	370	0
5/31/84	Effluent	104	85	0	0	0	0	0	172	0
6/4/84	Effluent	205	115	0	0	0	0	0	298	0
6/14/84	Effluent	78	373	0	0	0	10	12	393	0
6/17/84	Effluent	102	226	0	0	0	10	25	304	0
6/24/84	Effluent	55	0	0	0	0	0	0	55	0
6/28/84	Effluent	160	351	0	0	0	5	0	447	0
6/30/84	Effluent	152	108	0	0	0	0	3	241	0
7/3/84	Effluent	346	241	0	0	0	0	0	542	0
7/6/84	Effluent	106	117	0	0	0	0	0	201	0

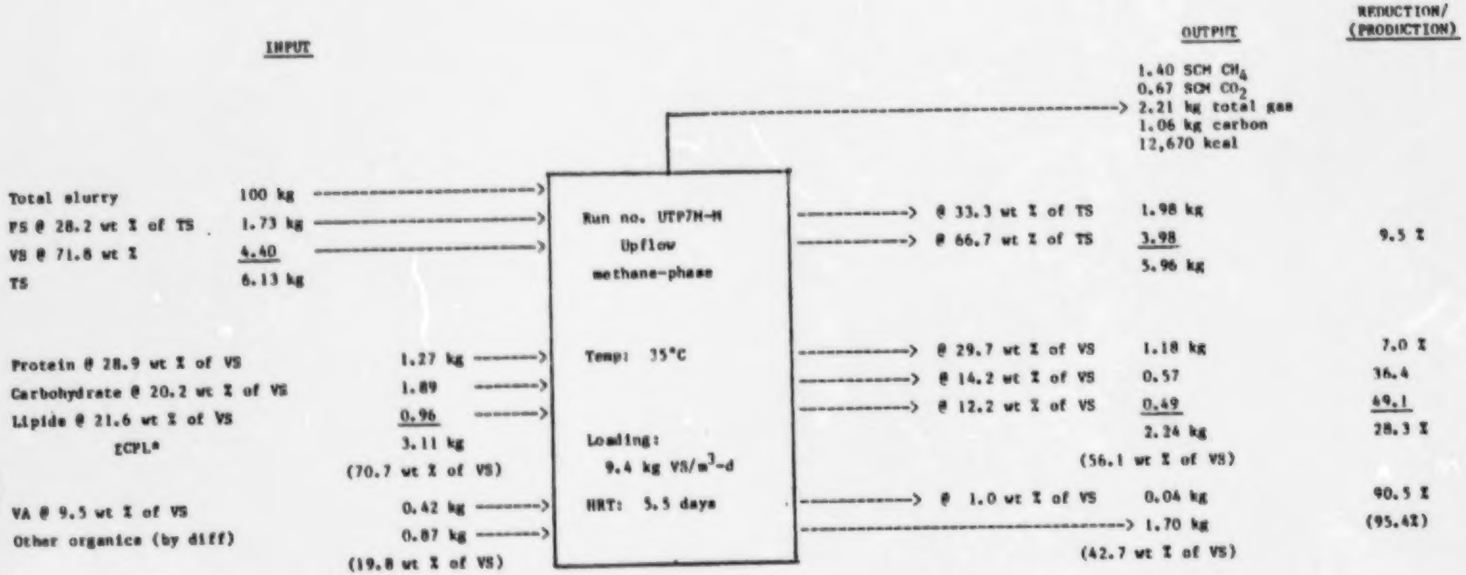
* This sample was taken from the bottom of the left-hand chamber (feed side) of the acid-phase digester.


INPUT
OUTPUT
REDUCTION/
(PRODUCTION)
Total slurry 100 kg
FS @ 25.2 wt % of TS 1.76 kg
VS @ 74.8 wt % 5.22
TS 6.98 kg
Protein @ 34.3 wt % of VS 1.79 kg
Carbohydrate @ 24.0 wt % of VS 1.25
Lipids @ 29.4 wt % of VS 1.53
ΣCPL* 4.57 kg
(87.6 wt % of VS)
VA @ 4.7 wt % of VS 0.25 kg
Other organics (by diff) 0.40 kg
(7.7 wt % of VS)
Feed, Q
Run no. UTP7M-M
Upflow
acid-phase
Temp: 35°C
Loading:
26.1 kg VS m^{3}-d
HRT: 2.0 days
0.43 SCM CH$_4$
0.31 SCM CO$_2$
0.88 kg total gas
0.38 kg carbon
3910 kcal
Overflow, 0.885 Q
Underflow 0.115 Q
Total
@ 28.6 wt % of TS 1.52 kg
@ 71.4 wt % of TS 3.80
5.32 kg
@ 26.1 wt % of TS 0.21 kg
@ 73.9 wt % of TS 0.60
0.81 kg
1.73 kg
4.40 (71.8 wt % of TS)
6.13 kg
15.7 %
@ 29.1 wt % of VS 1.11 kg
@ 19.8 wt % of VS 0.76
@ 21.5 wt % of VS 0.82
2.69 kg
(70.8 wt % of VS)
@ 27.5 wt % of VS 0.16 kg
@ 22.4 wt % of VS 0.13
@ 22.5 wt % of VS 0.13
0.42 kg
(70.0 wt % VS)
1.27 kg
0.89
0.95
3.11 kg
(70.5 wt % of VS)
29.0 %
29.0
37.8
32.0 %
@ 8.9 wt % of VS 0.34 kg
0.77 kg
@ 13.0 wt % of TS 0.08 kg
0.10 kg
0.42 kg
0.87 kg
(19.8 wt % of VS)
(40.5 %
(117.5%)

FS balance: effluent/feed = 97%
VS balance: effluent/feed = 101%
Carbon recovery in gas = 12%
Energy recovery in gas = 12%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure E-1. Mass balances for mesophilic upflow acid-phase digester for meso-meso upflow two-phase Run UTP7M-M conducted with Hanover Park sludge at a 7-day system HRT


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
1.40 SCM CH_4
0.67 SCM CO_2
2.21 kg total gas
1.06 kg carbon
12,670 kcal
Total slurry
100 kg
FS @ 28.2 wt % of TS
1.73 kg
VS @ 71.8 wt %
4.40
TS
6.13 kg
Run no. UTP7M-M
Upflow
methane-phase
@ 33.3 wt % of TS
1.98 kg
@ 66.7 wt % of TS
3.98
9.5 %
5.96 kg
Protein @ 28.9 wt % of VS
1.27 kg
Carbohydrate @ 20.2 wt % of VS
1.89
Lipids @ 21.6 wt % of VS
0.96
ΣCPL*
3.11 kg
(70.7 wt % of VS)
Temp: 35°C
Loading:
9.4 kg VS/m^3-d
HRT: 5.5 days
@ 29.7 wt % of VS
1.18 kg
7.0 %
@ 14.2 wt % of VS
0.57
36.4
@ 12.2 wt % of VS
0.49
49.1
2.24 kg
28.3 %
(56.1 wt % of VS)
VA @ 9.5 wt % of VS
0.42 kg
Other organics (by diff)
0.87 kg
(19.8 wt % of VS)
@ 1.0 wt % of VS
0.04 kg
90.5 %
1.70 kg
(95.4%)
(42.7 wt % of VS)

FS balance: effluent/feed = 114%

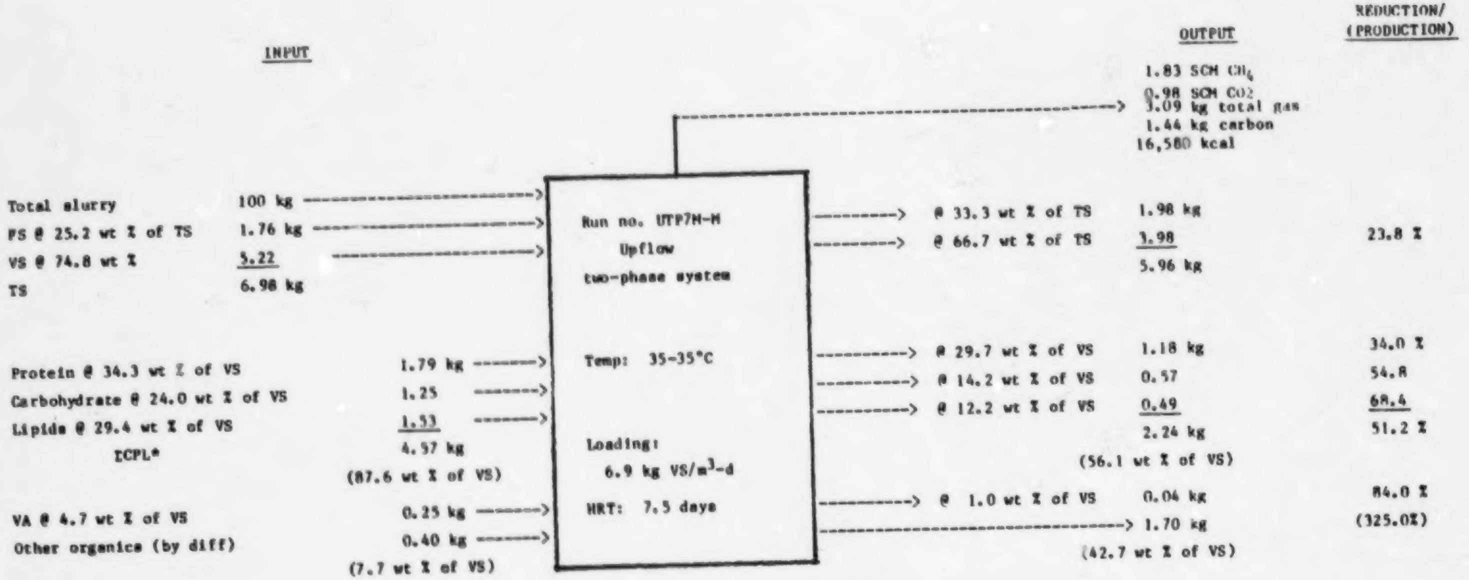
VS balance: effluent/feed = 141%

Carbon recovery in gas = 41%

Energy recovery in gas = 45%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure E-2. Mass balances for mesophilic upflow methane-phase digester for meso-meso upflow two-phase Run UTP7M-M conducted with Hanover Park sludge at a 7-day system HRT


INPUT
OUTPUT
REDUCTION/ (PRODUCTION)
1.83 SCM CH4
0.98 SCM CO2
3.09 kg total gas
1.44 kg carbon
16,580 kcal
Total slurry 100 kg
FS @ 25.2 wt % of TS 1.76 kg
VS @ 74.8 wt % 5.22
TS 6.98 kg
Run no. UTP7M-M
Upflow
two-phase system
@ 33.3 wt % of TS 1.98 kg
@ 66.7 wt % of TS 3.98 23.8 %
5.96 kg
Protein @ 34.3 wt % of VS 1.79 kg
Carbohydrate @ 24.0 wt % of VS 1.25
Lipids @ 29.4 wt % of VS 1.53
ΣCPL* 4.57 kg
(87.6 wt % of VS)
Temp: 35-35°C
Loading: 6.9 kg VS/m³-d
HRT: 7.5 days
@ 29.7 wt % of VS 1.18 kg 34.0 %
@ 14.2 wt % of VS 0.57 54.8
@ 12.2 wt % of VS 0.49 68.4
2.24 kg 51.2 %
(56.1 wt % of VS)
VA @ 4.7 wt % of VS 0.25 kg
Other organics (by diff) 0.40 kg
(7.7 wt % of VS)
@ 1.0 wt % of VS 0.04 kg 84.0 %
1.70 kg (325.0%)
(42.7 wt % of VS)

FS balance: effluent/feed = 112%

VS balance: effluent/feed = 135%

Carbon recovery in gas = 47%

Energy recovery in gas = 49%

* ΣCPL is the sum of protein, carbohydrate, and lipids.

Figure E-3. Mass balances for meso-meso upflow two-phase Run UTP7M-M conducted with Hanover Park sludge at a 7-day system HRT

TABLE E-3. NORMALIZED GAS COMPOSITIONS OBSERVED DURING RUN NO. UAP2M: UPFLOW MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
7/13/84	--	44.17	0.63	55.20
7/20/84	--	43.36	0.73	56.90
7/27/84	--	39.02	0.31	60.67
8/3/84	--	39.98	0.83	59.18
9/11/84	--	42.25	0.35	57.40
8/16/84	--	41.79	0.37	57.84
8/17/84	--	42.73	0.59	56.68

TABLE E-4. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING RUN NO. UAP2M: UPFLOW UPFLOW MESOPHILIC (35°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 2-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
7/12/84	1380	1569	289	522	362	420	48	3689	0
7/13/84	1147	1577	317	489	390	375	44	3448	0
7/20/84	1072	1405	155	372	294	284	24	2923	0
7/27/84	873	1652	271	282	207	180	44	2840	0
8/3/84	865	798	179	75	279	111	215	2025	0
8/10/84	1685	978	305	535	473	234	102	3519	0
8/17/84	1258	815	209	231	355	175	79	2571	0

TABLE E-5. NORMALIZED GAS COMPOSITIONS OBSERVED DURING RUN NO. UAP2T: UPFLOW THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 2-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
9/12/84	--	54.21	1.97	43.82
9/14/84	--	53.85	4.17	41.98
9/24/84	--	52.58	2.33	45.10
9/26/84	--	49.93	1.78	48.29

TABLE E-6. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING RUN NO. UAP2T: UPFLOW THERMOPHILIC (55°C) ACID-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE CONDUCTED AT A 2-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
9/12/84	2766	1591	642	827	1101	0	88	5750	0
9/14/84	3038	1829	658	918	1370	0	2	6401	0
9/19/84	1852	1117	285	571	493	0	0	3633	0
9/21/84	2411	1586	470	852	926	217	189	5367	42

TABLE E-7. NORMALIZED GAS COMPOSITIONS OBSERVED DURING
RON NO. UPT17M-T: MESO-THERMO TWO-PHASE DIGESTION OF
HANOVER PARK SLUDGE CONDUCTED AT A 17-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Run no. UAP4.5M)				
8/24/84	--	42.74	0.60	56.66
8/31/84	--	36.68	0.52	62.80
Methane-phase (Run no. UMP12T)				
8/24/84	--	46.30	0.00	53.70
8/31/84	--	44.06	2.04	53.89

TABLE E-8. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING RUN NO. TP17M-T: UPFLOW MESO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 17-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Run no. UAP4.5M)									
8/24/84	1511	1158	215	325	351	229	93	3207	0
8/31/84	580	794	18	0	98	0	115	1353	0
Methane-phase (Run no. UMP12T)									
8/24/84	1398	1291	226	135	546	0	87	3057	0
8/31/84	1924	1639	511	279	809	0	136	4337	0

TABLE E-9. NORMALIZED GAS COMPOSITIONS OBSERVED DURING RUN NO. UTP7T-T: UPFLOW THERMO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Run UAP2.1T)				
10/2/84	--	51.33	2.34	46.34
10/4/84	--	50.21	4.02	45.77
Methane-phase (Run UMP5.4T)				
9/29/84	--	45.17	1.52	53.31
10/5/84	--	46.12	3.28	50.60

TABLE E-10. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING RUN NO. UTP7T-T: UPFLOW MESO-THERMO TWO-PHASE DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 7-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Run UAP2.1T)									
9/29/84	1527	912	246	438	488	168	74	3156	39
10/2/84	1587	813	275	441	553	123	58	3163	13
10/4/84	1167	575	204	406	399	99	73	2379	12
10/5/84	1159	471	180	359	368	91	92	2226	0
Methane-phase (Run UMP5.4T)									
9/28/84	1495	1354	373	264	510	155	0	3370	55
10/2/84	1766	1251	339	463	632	148	7	3789	0
10/5/84	1965	1188	374	584	742	172	0	4118	0

TABLE E-11. NORMALIZED GAS COMPOSITIONS OBSERVED DURING RUN NO. UTP20T-T-T: UPFLOW/CFCSTR THERMO-THERMO-THERMO THREE-PHASE DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 20-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Upflow acid-phase (Run UAP1.9T)				
10/8/84	0.00	49.61	3.97	46.42
Upflow methane-phase (Run UMP5.2T)				
10/13/84	--	38.33	0.60	61.07
CFCSTR methane-phase (Run CMP13T)				
10/13/84	--	20.80	0.89	78.32

TABLE E-12. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING RUN NO. UTP20T-T-T: UPFLOW/CFCSTR THERMO-THERMO-THERMO THREE-PHASE DIGESTION OF HANOVER PARK SLUDGE CONDUCTED AT A 20-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Upflow acid-phase (Run UAP2.1T)									
10/13/84	803	414	153	188	268	8	46	1557	0
Upflow methane-phase (Run UMP5.2T)									
10/13/84	1345	685	235	227	410	43	73	2519	0
CFCSTR methane-phase (Run CMP13T)									
10/13/84	229	782	178	0	303	13	78	1211	0

TABLE E-13. NORMALIZED GAS COMPOSITIONS OBSERVED DURING STEADY-STATE RUN NO. TP3M-M(E): CFCSTR MESO-MESO TWO-PHASE DIGESTION OF ENZYMATICALLY PRETREATED, MIXED DOWNERS GROVE PRIMARY AND STICKNEY ACTIVATED SLUDGES CONDUCTED AT A 3-DAY HRT

Date	Hydrogen, mol %	Carbon dioxide, mol %	Nitrogen, mol %	Methane, mol %
Acid-phase (Digester no. 334)				
12/27/84	0.11	42.14	0.48	57.28
12/29/84	--	44.77	0.26	54.97
1/5/85	--	43.90	0.51	55.59
1/7/84	0.00	41.79	0.32	57.89
1/8/85	--	41.30	0.24	58.45
1/9/85	--	38.77	0.36	60.87
Methane-phase (Digester no. 333)				
12/27/84	--	34.70	0.00	65.30
12/29/84	--	34.30	0.00	65.70
1/5/85	--	34.72	0.00	65.28
1/7/85	--	34.43	0.00	65.57
1/8/85	--	33.49	0.00	66.51
1/9/85	--	32.72	0.00	67.28

TABLE E-14. VOLATILE ACIDS AND ETHANOL CONCENTRATIONS OBSERVED DURING STEADY-STATE RUN NO. TP3M-M(E): CFCSTR MESO-MESO TWO-PHASE DIGESTION OF ENZYMATICALLY PRETREATED, MIXED DOWNERS GROVE PRIMARY AND STICKNEY ACTIVATED SLUDGES CONDUCTED AT A 3-DAY HRT

Date	Acetic, mg/L	Propionic, mg/L	Isobutyric, mg/L	Butyric, mg/L	Isovaleric, mg/L	Valeric, mg/L	Caproic, mg/L	Total as acetic, mg/L	Ethanol, mg/L
Acid-phase (Digester no. 334)									
2/27/84	1,269	1,100	191	504	293	226	47	2,964	0
2/29/84	88	1,099	0	233	256	376	24	1,522	0
1/5/85	1,234	1,214	239	658	417	685	38	3,497	19
1/7/85	1,462	1,527	279	694	462	755	46	4,102	24
1/8/85	1,249	1,431	268	571	458	691	19	3,666	0
1/9/85	1,452	1,368	296	721	528	823	28	4,063	34
1/11/85	1,687	1,430	334	711	569	826	30	4,394	0
Methane-phase (Digester no. 333)									
12/27/84	188	751	0	0	0	0	7	800	0
12/29/84	77	895	0	0	27	0	34	836	0
1/5/85	202	1,029	33	0	74	31	22	1,131	0
1/7/85	238	1,270	21	0	97	22	22	1,364	0
1/8/85	274	1,058	62	0	79	22	0	1,233	0
1/9/85	257	1,004	52	30	75	27	0	1,187	32
1/11/85	168	838	19	0	68	18	0	911	0

TABLE E-15. VOLATILE SOLIDS AND ORGANIC COMPONENT CONCENTRATIONS AND REDUCTIONS OBSERVED DURING STEADY-STATE ADVANCED MESO-MESO TWO-PHASE DIGESTION OF HANOVER PARK SEWAGE SLUDGE*

Run	Sample	Sample date(s)[b]	Lot	Batch	TS	VS		Crude protein		Carbohydrates		Lipids		Organic reductions, %		
					mg/L	mg/L	wt % of TS	mg/L	wt % of VS	mg/L	wt % of VS	mg/L	wt % of VS	Protein	Carbohydrate	Lipids
UTP7M-M	Feed	6/28-7/1/84	13	1	69,870	52,230	74.75	17,456	33.42	11,956	22.89	--	--			
		6/28-7/1/84	13	1	69,870	52,230	74.75	--	--	13,934	26.68	--	--			
			Run means[c]		69,870	52,230	74.75		33.42		24.[illegible]		--			
			Feed means[d]						35.09		23.13		29.35			
			Final means[e]					17,894	34.26	12,514	23.96	15,330	29.35			
	Acid-phase effluent	6/28-7/1/84	13	1	61,310	44,010	71.78	--	--	8344	18.96	--	--			
		6/28-7/1/84	13	1	61,310	44,010	71.78	12,706	28.87	9621	21.47	9536	21.66			
					61,310	44,010	71.78	12,706	28.87	8881	20.18	9536	21.66	28.99	29.03	37.81
	Methane-phase effluent	6/28-7/1/84	13	1	59,640	39,790	66.72	11,819	29.70	5830	14.65	6849	12.19			
		6/28-7/1/84	13	1	59,640	39,790	66.72	--	--	5656	14.21	--	--			
		6/28-7/1/84	13	1	59,640	39,790	66.72	--	--	6303	15.86	--	--			
		6/28-7/1/84	13	1	59,640	39,790	66.72	--	--	4818	12.11	--	--			
					59,640	39,790	66.72	11,819	29.70	5650	14.20	6849	12.19	6.98	36.38	49.14
													System	33.95	54.85	60.37
TP3M-M[f]	Feed	1/8-1/12/85	23	1	69,470	46,530	68.26	--	--	15,734	21.80	9370	20.14			
			Run means		69,470	46,530	68.26		--		21.80		20.14			
			Feed means						--		21.80		20.14			
			Final means					--	--	15,734	21.39	9370	19.76			
	Acid-phase effluent	1/8-1/12/85	23	1	60,410	39,170	64.84	--	--	7882	20.12	8990	22.95			
					60,410	39,170	64.84	--	--	7882	20.12	8990	22.95	--	49.90	4.06
	Methane-phase effluent	1/8-1/12/85	33	1	56,900	33,820	61.60	--	--	5729	16.94	5729	16.94			
					56,900	33,820	61.60	--	--	5729	16.94	5729	16.94	--	27.32	36.27
													System	--	63.59	38.86

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* Data reported are the averages of duplicate or triplicate determinations.

[b] A single sample date indicates that the analyses were conducted on a grab sample collected that day. A time period under this column indicates the start and end dates of collection of a grab or time-composite sample used for the analyses.

[c] Run means are the averages of the feed analyses conducted for a particular steady-state run on a single feed lot and batch.

[d] Feed means are the average organic contents (expressed as weight percent of VS) of all steady-state samples collected for a particular feed lot and batch.

[e] Final means are the average feed slurry organic concentrations and contents used to determine the organic reductions. The organic contents are the average of the feed and run organic contents. Organic feed concentrations were calculated as the product of the final mean organic feed contents and the average feed volatile solids concentration for the run.

[f] Steady-state run TP3M-M(E) was conducted with mixed Downers Grove primary and Stickney activated sludges. The feed was treated for 24 hrs at 35°C with cellulase (Novo Celluclast 1.5L) and cellobiase (Novozym 188) enzymes at dosages of 2.75 g/kg feed TS and 0.28 g/kg feed TS, respectively. Lipase (Novozym 225) was added to the acid digester at a dosage of 2.75 g/kg feed TS.

APPENDIX F

FEED SLUDGE LOTS AND BATCHES USED DURING STEADY-STATE DIGESTION RUNS

TABLE F-1. FEED SLUDGE LOTS AND BATCHES USED DURING STEADY-STAGE DIGESTION RUNS

Run number	Feed sludge lot/batch number*
SS15M	1/8-9
SS7M	6/1-3
SS3M	8/5
SS15T	12/1
SS7T	16/1
SS3T	16/1
TP15M-M	5/3-4
TP7M-M	17/1
TP3M-M	28/1
TP3M-M(E)	32/1
TP15M-T	16/1
TP7M-T	17/1
TP7T-T	18/5-7
TP3T-T	19/3-6
UTP7M-M	13/1
AP2M7	6/2-3
AP2M6	12/1
AP2M5.5	12/1
AP2M5	13/1
AP1.3M7	8/5
AP1.3M5	13/1
AP2T7	5/3-4
AP2T6	12/1
AP2T5.5	12/1
AP2T5	13/1
AP1.3T7	8/5
AP1.3T5	13.1

* Analyses of these feed sludges are shown in Appendix A, Tables 1-6.

APPENDIX G

COMPARISON OF CALCULATION OF VOLATILE SOLIDS BY MOP-16 FORMULA WITH MATERIAL BALANCE METHOD

TABLE G-1. COMPARISON OF CALCULATION OF VOLTAILE SOLIDS REDUCTION BY MOP-16 FORMULA WITH MATERIAL BALANCE METHOD[1]

	Volatile solids reduction				
Run number	Material balance[2]	MOP-16[3]	Difference	Percent loss in ash relative to total solids[4]	VS reduction (Wt.-of-gas method)[5]
SS15M	33.0	38.2	-5.2	-2.17	28.8
SS7M	27.3	18.3	5.0	2.07	32.7
SS3M	15.6	13.7	1.9	0.51	19.3
SS15T	41.3	36.8	4.5	1.71	45.9
SS7T	31.0	32.9	-1.9	-0.74	39.6
SS3T	12.1	19.0	-6.9	-2.27	20.2
TP15M-M	34.0	37.2	-3.2	-0.55	63.4
TP7M-M	32.8	33.6	-0.8	-0.20	51.5
TP3M-M	24.8	26.5	-1.7	-0.38	35.5
TP15M-T	38.2	28.0	10.2	1.92	48.5
TP7M-T	34.3	32.8	1.5	0.41	54.7

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[1] Data used are from Tables B-13 and C-15.

[2] Material balance: Assumes volume in = volume out,

$$VS_R = \frac{\text{mg VS/L in feed} - \text{mg VS/L in effluent}}{\text{mg VS/L in feed}} \times 100$$

[3] MOP-16: Assumes ash in = ash out,

$$VS_R = \frac{VS_i - VS_o}{VS_i - (VS_i \times VS_o)} \times 100$$

where $VS_{i,o}$ = VS as fraction of TS at influent and effluent

[4] Ash Loss: $\% \text{ Loss} = \frac{\text{mg ash/L in feed} - \text{mg ash/L in effluent}}{\text{mg TS/L in feed}} \times 100$

[5] VS reductions were taken from Tables 49, 52, 57, and 60.